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SAMPLING AND ANALYSIS PLAN FOR REMEDIAL INVESTIGATION SITE 2 WORLD WAR II  
LANDFILL NCBC GULFPORT FL  
4/1/2012  
TETRA TECH

# Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



Rev. 1  
04/19/12

## Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation at Site 2 – World War II Landfill

Naval Construction Battalion Center Gulfport  
Gulfport, Mississippi

Contract Task Order 0150

April 2012



NAS Jacksonville  
Jacksonville, Florida 32212-0030



Document Tracking Number 12JAX0096

April 19, 2012

Project Number 112G02094

Commanding Officer, Southeast  
Naval Facilities Engineering Command  
Attn: Charles Cook (Code OPA6)  
Remedial Project Manager  
NAS Jacksonville  
135 Ajax Street  
Jacksonville, Florida 32213-0030

Reference: CLEAN IV Contract Number N62467-04-D-0055  
Contract Task Order Number 0150

Subject: Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Remedial Investigation at Site 2  
Naval Construction Battalion Center Gulfport, Mississippi

Dear Mr. Cook:

Tetra Tech is pleased to submit the Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Remedial Investigation at Site 2 at Naval Construction Battalion Center (NCBC) Gulfport along with the Response to Comments letter and the living compact disc (CD) for CTO 0150.

If you have any questions with regard to this submittal, please contact me via e-mail at [Gregory.Roof@TetraTech.com](mailto:Gregory.Roof@TetraTech.com) or by phone at (904) 730-4669, extension 215.

Sincerely,



Gregory S. Roof, P.E.  
Task Order Manager

GSR/lc

c: Gordon Crane, NCBC Gulfport (2 hardcopies, 1 CD)  
Bob Merrill, MDEQ (1 hardcopy, 1 CD)  
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April 19, 2012

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Reference: CLEAN IV Contract Number N62467-04-D-0055  
Contract Task Order Number 0150

Subject: Response to Comments, Draft-Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Remedial Investigation at Site 2  
Naval Construction Battalion Center Gulfport, Mississippi

Dear Mr. Cook:

Tetra Tech is pleased to submit this letter responding to the comments from the Mississippi Department of Environmental Quality (MDEQ) on the Draft-Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for the Remedial Investigation at Site 2 at Naval Construction Battalion Center (NCBC) Gulfport. The questions and/or comments received by Tetra Tech are addressed below.

**MDEQ, Mr. Bob Merrill**

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**Comment 1:** Contact (telephone) information (page 12) from the state RPM is incorrect; 961-5302 should read 961-5049 (for Bob Merrill).

**Response:** The Final Sampling and Analysis Plan (SAP) was updated using the correct telephone number.

**Comment 2:** The acronym list (pages 5 through 10) does not identify the acronyms SSL or R5 ESL.

**Response:** The SAP was updated and the acronyms were identified.

**Comment 3:** Clarification is needed in the text discussion concerning previous dioxin groundwater occurrences at Site 2 and northerly adjoining Site 7. The dates of investigations and the identity and location of monitor wells located near or at sites 2 and 7 are not given in text discussion presented on page 32. Dioxin concentrations above groundwater regulatory screening levels were (apparently) reported from samples collected during these investigations. The location of monitoring well GPT-2-3 (for

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which no specific concentration is given) is described (page 32, paragraph 1) as “near of Site 7 and just north of Site 2”. The following paragraph (page 32) describes “an additional investigation” during which “one monitoring well at Site 7 contained 51.6 pg/L dioxin with an estimated 25 pg/L attributed to TCDD”, but the dates of the investigations and identities of the wells with TCDD exceedances are not clearly correlated with Site 2.

**Response:** Additional information and figures on the location of the monitoring wells and the sampling dates were added to the updated SAP in Sections 10.2.3 and 10.2.4.

**Comment 4:** Concerning the text statement (page 32, paragraph 2) regarding the amount of 2,3,7,8 TCDD in the referenced groundwater sample (25 ppt); it should be noted that OPC does not exclusively evaluate 2,3,7,8 TCDD because several of the associated (tetra-type) congeners are also of importance in the evaluation of suspected occurrences of and attribution to Herbicide Orange (HO). Dioxin screening values among the various media utilize the total of congener concentrations (TEF values) to establish the TEQ screening value used. The occurrence or lack of TCDD is a good indicator of the presence of HO but the MCL (30.0 ppq) addresses the sum of all congeners in the sample (51.6 ppq) and not just the 25 ppq TCDD congener concentration, so the sample did exceed the MCL and the presence of HO in areas within or adjoining the site is established. This should be clearly stated in an expanded text providing support for the decision to include dioxin analyses among the various media.

**Response:** The reference to the “non-HO related dioxin” related to groundwater results associated with Site 5 (Harding Lawson Associates, 1999). Site 5 is over 2,000 feet to the southwest of Site 2 and is located on the southeastern side of a groundwater divide that isolates Site 5 from Site 2 (Plate 3 in Appendix B of the SAP). The “51.6 picograms per liter (pg/L) dioxin with an estimated 25 pg/L attributed to TCDD” was in reference to the values associated with the groundwater analytical results from the Site 7 monitoring well GPT-7-1. Site 7 is located adjacent to and north of Site 2, and monitoring well GPT-7-1 is located approximately 100 feet north of Investigation Boundary for Site 2. Figure 6A in the updated SAP displays the location of monitoring well GPT-7-1 in relation to Site 2. The text in the updated SAP no longer mentions Site 5 or the “non-HO related dioxin” as neither Site 5 nor “non-HO related dioxin” is the focus of the sampling activities at Site 2. Additionally, Section 10.4.1 “Sources and Potential Contaminants” of the SAP was updated to provide support for the decision to include dioxin analyses among the various media.

**Comment 5:** The sampling plan (Table 15) indicates that dioxin analyses will be completed for soil (page 57), sediment (page 66) and groundwater (page 75) but not for surface water. The text should clarify why dioxin analysis is not planned for surface water.

**Response:** There are two reasons why dioxin analysis is not planned for surface water. One relates to the history of the site and the other relates to the use of the data.

- From the historical perspective: The Site 2 landfill was operated and closed before HO was stored on the base. In addition, the ditches on the eastern side of the site were all excavated as part of the remedy for Site 8. The pond, where the sediment samples are planned to be collected, was dug well after HO storage ended and does not receive any surface water from the base drainage system.
- From the data usage perspective: Sediments sample analysis serves as a better indicator of contamination than do surface water samples. Dioxins have a very high octanol/water partition coefficient and a very low solubility limit. Therefore, dioxins adhere to sediments and do not enter into the water column at readily detectable concentrations. The general transport mechanism for dioxins in streams and ditches is through sediment transport and/or sediment entrainment in the water column. Such entrainment typically occurs during high velocity stream water flow events which often follow heavy rainfall events.

The population of interest for sediments includes any sediment along the western shoreline of the pond that may be impacted by contaminated groundwater that potentially migrates from the site and recharges the pond (see Figure 6 in the SAP). Therefore, given the history of Site 2, the history of the pond, and the chemical/physical properties of dioxin, it was determined that analyzing the sediments for dioxin better met the project data quality objectives than analyzing the surface water for dioxin.

**Comment 6:** A surface sheen (rainbow colors) and distressed vegetation were observed in surface water drainage ditches located along the south and east sides of Site 2 (Appendix B) in December of 1994. Severely distressed vegetation (dead trees) was observed in areas adjoining the ditches. These observations should be addressed in the sampling program (ex. Soil, sediment and surface water sampling and analyses for TPH, PAHs, VOCs, SVOCs and possibly dioxin) if the surface water sheen and distressed vegetation are still apparent. These observations (included in Appendix B) and associated decisions addressing the possible contamination in these areas should be discussed in the main body of the text.

**Response:** The one-page December 22, 1994, memorandum with the attached one-page hand drawing from Ms. Penny Baxter (ABB) to Mr. Art Conrad (Southern Division Naval Facilities Engineering Command) references the sheen and the distressed vegetation. Figure 6A depicting the estimated location of the area of former distressed vegetation and ditch with sheen has been added to the updated SAP. The ditch was filled in during golf course construction activities, and a concrete culvert was installed in the location of the former ditch to facilitate drainage. To assess potential impacts to the soil and groundwater in the location of the distressed vegetation and drainage ditch, soil and groundwater samples will be collected in these areas. Figure 6A also depicts the locations of the soil and groundwater samples that have been added to the SAP.

**Comment 7:** Clarification is needed concerning planned sampling activities in areas along the eastern site boundary and how data gaps and field observations from previous studies (referenced in the sampling plan) will be addressed, as no sampling locations in these previously unsampled areas are shown on Figure 6. Three groundwater samples and one surface water/sediment collocated sample are described in the Verification Study (1988, Table 8) included in Appendix B, however the location of the surface water/sediment sample and the association with Site 2 is not apparent. No sample locations (for the groundwater, surface water or sediment media) are shown for eastern areas of the site as the groundwater sampling locations (two of which are located at Site 2) are in the southern portion of Site 2 and one (GPT 2-3) is located at the northern boundary of northerly adjoining Site 7 (1988, Plate 3).

The text discussion reference Appendix B (page 31, paragraphs 2 through 5) should (at least generally) specify locations of referenced (1988) surface water/sediment samples (discussed on page 31, paragraph 3) and be expanded to demonstrate how the planned sampling strategy addresses data gaps resulting from contaminated areas reported during previous field observations (technical memorandum dated 22 December 1994) and sampling investigations (Verification Study dated 17 July 1988).

**Response:** Comment 7 addresses more than one topic; therefore, this response is broken down into several sections.

- Regarding clarification of planned sampling activities and how data gaps and previous field observations will be addressed: The SAP presents a flexible and iterative approach to sampling. Fieldwork for the RI consists of four events; i.e.; (1) Geophysical Survey, (2) Passive Soil Gas Survey, Landfill Gas Survey, Ditch and Pond Investigation, (3) Soil and Groundwater Sampling, and (4) Monitoring Well Installation and Additional Sampling as Needed. Each event provides information that will be used in the next event to refine the location, number, and of type of sample collection points. For example, during Event 2, it is anticipated that 49 GORE-SORBER® Modules will be installed in a grid pattern over Site 2 (see Figure 6 in the SAP). The locations of the soil and

groundwater samples in Event 3 will be based upon the results from the GORE-SORBER® Modules and the Event 1 geophysical survey. Therefore, it is not possible at this time to show all the anticipated soil and groundwater locations. As explained in the Response to Comment 6, additional sample locations for soil and groundwater are included to assess the former area of the distressed vegetation and ditch containing the sheen as reported in the one-page December 22, 1994, memorandum by Ms. Penny Baxter (ABB). The actual number of soil and groundwater samples may increase or decrease based upon the findings from other events in this investigation. Worksheets 14 and 17 of the SAP were updated to clarify the iterative approach to sampling and how the results from one event will aid in determining the sample location points referenced in later events. In addition, Worksheet 14 of the SAP was updated to note that the Project Manager will provide information to the Project Team at the end of Events 1, 2, and 3 that summarized the findings and how those findings will be used to shape the activities planned for the next event.

- Regarding surface water/sediment samples discussed on page 31, paragraph 3: These samples were collected from the drainage ditch near the southeastern side of the intersection of Colby Avenue and 8<sup>th</sup> Street. More details and locations are provided in Section 10.4 of the updated SAP. The samples were analyzed for selected metals (cadmium, chromium, and lead), oil and grease, total organic carbon, total organic halides, and chemical oxygen demand. Low levels of chromium and lead were detected below regulatory levels in the sediment sample. Other metal concentrations were less than the laboratory detection limits. The text in the SAP was updated, and Plate 6 from the Verification Study was added to in Appendix B in the SAP.
- Expanded text in Worksheet 10 to demonstrate how the planned sampling strategy addresses data gaps: The Conceptual Site Model is presented in pages 30 through 34 of the SAP (Worksheet 10). The rationales for the sampling activities are presented in Worksheet 17, which was updated to demonstrate how the planned sampling strategy addresses data gaps resulting from contaminated areas reported during previous field observations.

**Comment 8:** Since the site hydrological setting is not fully understood, the groundwater sampling program should be more open ended than to plan limitations on the number (18) and depth (40 feet) of groundwater samples in the event that the area of influence (or the plume size) is larger than anticipated. Groundwater monitor wells should be located upgradient and downgradient of the site in an array that will define the plume.

Text discussions concerning the groundwater sampling strategy need to be expanded to clarify how the vertical and horizontal extent of the groundwater plume will be defined. The predetermined vertical boundary of investigation of approximately 40 feet of depth (page 38, paragraphs 3 and 4) will be invalid in the absence of a naturally occurring aquitard or aquiclude that will prevent downward contamination migration if DNPL contaminants (“sinkers”) such as TCE are present. The lateral extent of contamination should be defined by areas in which observed groundwater contaminants are no longer detected, although the text places limitations on the number of laterally located samples and states that the lateral extent of investigation will terminate at site boundaries if subsurface soil and/or groundwater do not exceed PALs (page 39, paragraph 5). The total planned number of groundwater samples is defined as “not to exceed 18 groundwater samples” (page 47, paragraph 3).

Several permanent (sentry) monitor wells should be established outside of the known areas of contamination (once determined) in the event that the stepped sampling strategy originating within the landfill does not provide an accurate conceptual site model.

**Response:** One goal of the SAP is to present a flexible and iterative sampling design. The flexibility in the design and the iterative nature of the approach will enable the Project Team to make adaptive management decisions that allow for acquisition of the type of data referenced in Comment 8. For example, matters related to the vertical and horizontal extent of the groundwater plume will be part of the



focus when monitoring wells are installed. The updated SAP notes that locations and depths of the investigation wells will depend upon the results of the earlier investigation events.

The installation of permanent monitoring wells, also known as post-closure monitoring wells, is one of the elements in the presumptive remedy. The suggested location, number, and depths of these monitoring wells will be presented in the Feasibility Study.

**Comment 9:** The document does not contain a Health and Safety Plan.

**Response:** The HASP is a stand-alone document. It was submitted as a final document in June 2009.

**Comment 10:** Clarification is needed concerning plans for conducting a human health risk assessment at Site 2. The document contains a detailed methodology for an Ecological Risk Assessment (Appendix C) but human health risk is only briefly addressed on page 40 (paragraph 2).

Although the text briefly discusses acceptable human health risk values (cancer 1E-6 and hazard quotient of 1.0) no plan is presented that will demonstrate actual risk at Site 2. The methodology for conducting a human health risk assessment should be included in the report of clarification of reasons to exclude it should be provided.

**Response:** The updated SAP now contains a detailed methodology for a Human Health Risk Assessment (see Appendix C).

If you have any questions with regard to this submittal, please contact me via e-mail at Gregory.Roof@TetraTech.com or by phone at (904) 730-4669, extension 215.

## REFERENCES

Harding Lawson Associates, 1999. Groundwater Monitoring Report Naval Construction Battalion Center Gulfport, Mississippi Unit Identification No.: N62604 Contract No.: N62467-89-D-0317/150 Prepared by: Harding Lawson Associates December 1999

Sincerely,



Gregory S. Roof, P.E.  
Task Order Manager

GSR/lc

c: Gordon Crane, NCBC Gulfport  
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CTO 0150 Project File

**SAP Worksheet #1 – Title and Approval Page**  
(UFP-QAPP Manual Section 2.1)

**SAMPLING AND ANALYSIS PLAN  
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)  
FOR  
REMEDIAL INVESTIGATION  
AT  
SITE 2 – WORLD WAR II LANDFILL**

**NAVAL CONSTRUCTION BATTALION CENTER GULFPORT  
GULFPORT, MISSISSIPPI**

**COMPREHENSIVE LONG-TERM  
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT**

**Submitted to:  
Naval Facilities Engineering Command  
Southeast  
NAS Jacksonville  
Jacksonville, Florida 32212-0030**

**Submitted by:  
Tetra Tech  
661 Andersen Drive  
Foster Plaza 7  
Pittsburgh, Pennsylvania 15220**

**CONTRACT NUMBER N62467-04-D-0055  
CONTRACT TASK ORDER 0150**

**APRIL 2012**

**SAP Worksheet #1 – Title and Approval Page**  
(UFP-QAPP Manual Section 2.1)

**Document Title:** Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation at Site 2 - World War II Landfill, Naval Construction Battalion Center Gulfport, Mississippi

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**Preparer's Name and Organizational Affiliation:** Yarissa Martínez and Peggy Churchill, Tetra Tech

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Jonathan Tucker  
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Approval Signature:

Approval was obtained via separate letter.  
\_\_\_\_\_  
Signature/Date  
Bob Merrill  
Mississippi Department of Environmental Quality

**SAP Worksheet #1 – Title and Approval Page**  
**(UFP-QAPP Manual Section 2.1)**

**Document Title:** Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation at Site 2 - World War II Landfill, Naval Construction Battalion Center Gulfport, Mississippi

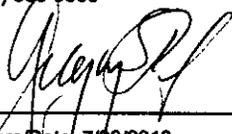
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## EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) encompasses Field Sampling Plan and Quality Assurance Project Plan requirements for a Remedial Investigation (RI) Work Plan at Site 2 – World War II Landfill at Naval Construction Battalion Center (NCBC) Gulfport, Mississippi. This document constitutes the planning document, addressing specific protocols for sample collection, sample handling and storage, chain-of-custody, laboratory and field analyses, data validation, and data reporting.

This SAP has been prepared by Tetra Tech on behalf of Naval Facilities Engineering Command Southeast under the Comprehensive Long-term Environmental Action Navy Contract Number N62467-04-D-0055, Contract Task Order 0150. This SAP was generated for and complies with applicable United States Navy, Mississippi Department of Environmental Quality (MDEQ), and United States Environmental Protection Agency (USEPA) Region 4 requirements, regulations, guidance, and technical standards. This includes the Department of Defense (DoD), Department of Energy (DoE), and USEPA Interagency Task Force environmental requirements regarding federal facilities. To comply with DoD/DoE/USEPA requirements, this SAP is presented in the format of standard worksheets specified in the Uniform Federal Policy for Quality Assurance Plans guidance document (USEPA, 2005).

NCBC Gulfport is located in the western part of Gulfport, Mississippi, in the southeastern part of Harrison County; about 2 miles north of the Gulf of Mexico (see Figure 1). The property for the installation was acquired in April 1942 and occupies approximately 1,100 acres. Nine sites at NCBC Gulfport, including Site 2, were identified in the Initial Assessment Study as potential threats to human health or the environment (Envirodyne Engineers, Inc., 1985). Six of the nine sites are former landfills, and Site 2 is the fifth former landfill at NCBC Gulfport to have an RI initiated. The identified sites are being investigated following the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) process with the MDEQ as lead regulatory agency.

The primary mission of NCBC Gulfport is to support military readiness for four battalions of the Naval Construction Force (NCF) and the storage and maintenance of pre-positioned War Reserve Material Stock. The NCF support consists of mobilization and logistics support for both homeport services and deployed support. Approximately 5,000 military and 1,600 civilian personnel are assigned to or employed by the NCBC Gulfport.

Site 2, referred to as the World War II Landfill, encompasses approximately 9.3 acres north of the intersection of 8<sup>th</sup> Street and Colby Ave (see Figure 2). The site operated as a landfill from 1942 until 1948. During this time, nearly all of the solid waste and some liquid and chemical waste generated at the facility were disposed of at this site. The site is currently used as a fairway for the Pine Bayou Golf

Course operated by NCBC Gulfport. As a result, it is understood and accepted by the Project Team that the Presumptive Remedy for CERCLA Municipal Landfill Sites as prescribed in the USEPA guidance document (USEPA, 1993) will be applied to the site. The selection of the Presumptive Remedy is supported by the Site 2 Conceptual Site Model and the data gathered during this streamlined RI/Feasibility Study (FS). The use of the streamlined RI/FS was developed by USEPA as a framework for the Presumptive Remedy. The Presumptive Remedy includes containment of the landfill contents and prevention of contaminant migration in the future. The primary objectives for the Site 2 RI are as follows:

- determine the previous landfill boundaries,
- evaluate contaminants are migrating from the site along with nature and extent of any contamination, if present, and
- gather site data that will be useful in developing an effective landfill cover for containment.

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## **LIST OF APPENDICES**

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- Appendix B: Historical Background Information
- Appendix C: Human Health Risk Assessment Methodology and Ecological Risk Assessment Methodology
- Appendix D: Field SOPs and Field Forms
- Appendix E: Analytical Laboratory SOPs and ELAP Certification for the Labs

## ACRONYMS

%D	Percent Drift
%R	Percent Recovery
AES	Atomic Emission Spectroscopy
APPL	APPL, Inc.
BFB	Bromofluorobenzene
bls	Below Land Surface
BNA	Base/Neutral/Acid
BSCEM	Baseline Site Conceptual Exposure Model
BTEX	Benzene, Toluene, Ethylbenzene, and Xylenes
°C	Degrees Celsius
CA	Corrective Action
CAS	Chemical Abstracts Service
CCC	Continuing Calibration Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
CSM	Conceptual Site Model
CTO	Contract Task Order
DCE	Dichloroethene
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
Eco-SSL	Ecological Soil Screening Level
ELAP	Environmental Laboratory Accreditation Program
EM	Electromagnetic
Empirical	Empirical Laboratories, LLC
ESV	Ecological Screening Value
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GC/ECD	Gas Chromatography Electron Capture Detector

## ACRONYMS (CONTINUED)

GC/MS	Gas Chromatography Mass Spectrometry
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HLA	Harding Lawson Associates
HO	Herbicide Orange
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDW	Investigation Derived Waste
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LUC	Land Use Control
µg/L	Microgram per Liter
MDEQ	Mississippi Department of Environmental Quality
mg/kg	Milligram per Kilogram
mL	Milliliter
MPC	Measurement Performance Criteria
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NA	Not Applicable
NaOH	Sodium Hydroxide
NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast
NCBC	Naval Construction Battalion Center
NCF	Naval Construction Force
PAH	Polynuclear Aromatic Hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyl
PCE	Tetrachloroethylene

## ACRONYMS (CONTINUED)

PCDD	Polychlorinated Dibenzodioxin
PCDF	Polychlorinated Dibenzofuran
pg/g	Picogram per Gram
pg/L	Picogram per Liter
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PWD	Public Works Division
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
R5 ESL	USEPA Region 5 RCRA Ecological Screening Level for Soil
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SSL	Soil Screening Level
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCDD	Tetrachlorodibenzo-p-dioxin
TCE	Trichloroethylene
TOC	Total Organic Carbon
TOM	Task Order Manager
TPH	Total Petroleum Hydrocarbon

### ACRONYMS (CONTINUED)

TRG	Target Remediation Goal
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
VLF	Very Low Frequency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

**SAP Worksheet #2 – SAP Identifying Information**  
 (UFP-QAPP Manual Section 2.2.4)

**Site Name/Number:** Naval Construction Battalion Center (NCBC), Gulfport, Mississippi  
**Operable Unit:** Site 2 – World War II Landfill  
**Contractor Name:** Tetra Tech  
**Contract Number:** N62467-04-D-0055  
**Contract Title:** Comprehensive Long-term Environmental Action Navy (CLEAN)  
**Work Assignment Number** Contract Task Order (CTO) 0150

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (United States Environmental Protection Agency [USEPA], 2005) and *Guidance for Quality Assurance Project Plans, QA/G-5, QAMS* (USEPA, 2002).
2. Identify regulatory program: National Contingency Plan; Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)
3. This SAP is a project-specific SAP.
4. List dates of scoping sessions that were held:

SCOPING SESSION	DATE
Data Quality Objective (DQO) Meeting	05/13/2009

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

TITLE	DATE
None	

6. List organizational partners (stakeholders) and connection with lead organization:

Mississippi Department of Environmental Quality (MDEQ) (lead regulatory stakeholder)  
NCBC Gulfport (property owner)  
USEPA Region IV (regulatory stakeholder)

7. Lead organization

Naval Facilities Engineering Command Southeast (NAVFAC SE)

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Not Applicable (NA). There are no exclusions.

**SAP Worksheet #3 – Distribution List**  
 (UFP-QAPP Manual Section 2.3.1)

NAME OF SAP RECIPIENTS	TITLE/ROLE	ORGANIZATION	TELEPHONE NUMBER	E-MAIL ADDRESS OR MAILING ADDRESS	DOCUMENT CONTROL NUMBER
Robert Fisher	NAVFAC SE Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC SE Naval Air Station (NAS) Jacksonville Building 903 Jacksonville, FL 32212	(904) 542-6827	<a href="mailto:robert.r.fisher@navy.mil">robert.r.fisher@navy.mil</a>	NA
Gordon Crane	NCBC Gulfport Point of Contact (POC)/ Environmental Coordinator	NCBC Gulfport 2401 Upper Nixon Avenue Gulfport, MS 39501	(228) 871-2485	<a href="mailto:gordon.crane@navy.mil">gordon.crane@navy.mil</a>	NA
To Be Determined (TBD)	Head of Reference Desk (NCBC Gulfport Administrative Record)	TBD	TBD	TBD	NA
Mike Singletary	Environmental Engineer/ Environmental Technical Support	NAVFAC SE NAS Jacksonville Building 903 Jacksonville, FL 32212	(904) 542-6303	<a href="mailto:michael.a.singletary@navy.mil">michael.a.singletary@navy.mil</a>	NA
Bob Merrill	MDEQ RPM/ Provides Regulator Input	MDEQ 515 E Amite Street Jackson, MS 39201-2709	(601) 961-5049	<a href="mailto:bob_merrill@deg.state.ms.us">bob_merrill@deg.state.ms.us</a>	NA
Paul Necaïse	Ecologist/ Environmental Support	United States Fish and Wildlife Services 6578 Dogwood View Parkway Jackson, MS 39213	(228) 493-6631	<a href="mailto:paul_necaïse@fws.gov">paul_necaïse@fws.gov</a>	NA
TBD	USEPA RPM*/ Receive Final Document	USEPA Region 4 Atlanta Federal Center 61 Forsyth Street, SW Atlanta, GA 30303-8960		<u>TBD</u>	NA
Jon K Overholtzer	CH2M Hill Project Manager (PM)/ Partnering Team Member	Northpark 400 1000 Abernathy Road Suite 1600 Atlanta, GA 30328	(678) 530-4262	<a href="mailto:joverhol@ch2m.com">joverhol@ch2m.com</a>	NA

NAME OF SAP RECIPIENTS	TITLE/ROLE	ORGANIZATION	TELEPHONE NUMBER	E-MAIL ADDRESS OR MAILING ADDRESS	DOCUMENT CONTROL NUMBER
Debra Humbert	Tetra Tech Program Manager/ Manages Navy Initiatives	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-1990	<a href="mailto:debra.humbert@tetrattech.com">debra.humbert@tetrattech.com</a>	NA
Gregory Roof	Tetra Tech Task Order Manager (TOM)/ Manages Project Activities	Tetra Tech 8640 Philips Highway, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Ext 215	<a href="mailto:gregory.roof@tetrattech.com">gregory.roof@tetrattech.com</a>	NA
William Olson	Tetra Tech Field Operations Leader (FOL)/ Site Safety Officer (SSO)/ Manages Field Operation and Site Safety Issues	Tetra Tech 1558 Village Square Boulevard Suite 2 Tallahassee, FL 32309	(850) 385-9899 Ext 1359	<a href="mailto:william.olson@tetrattech.com">william.olson@tetrattech.com</a>	NA
Kelly Carper	Tetra Tech Quality Assurance (QA) Manager (QAM)/ Manages Corporate QA Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-7273	<a href="mailto:kelly.carper@tetrattech.com">kelly.carper@tetrattech.com</a>	NA
Matt Kraus	Tetra Tech Project Chemist/ Provides Coordination with Laboratories	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8729	<a href="mailto:matt.kraus@tetrattech.com">matt.kraus@tetrattech.com</a>	NA
Joseph Samchuck	Tetra Tech Data Validation Manager (DVM)/ Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8510	<a href="mailto:joseph.samchuck@tetrattech.com">joseph.samchuck@tetrattech.com</a>	NA
Matt Soltis	Tetra Tech Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8912	<a href="mailto:matt.soltis@tetrattech.com">matt.soltis@tetrattech.com</a>	NA
Kim Kostzer	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical Laboratories, LLC (Empirical) 621 Mainstream Drive Suite 270 Nashville, TN 37228	(615) 345-1115	<a href="mailto:kkostzer@empirilabs.com">kkostzer@empirilabs.com</a>	NA

\*USEPA involvement in NCBC Gulfport has been limited to requesting final documents.

NAME OF SAP RECIPIENTS	TITLE/ROLE	ORGANIZATION	TELEPHONE NUMBER	E-MAIL ADDRESS OR MAILING ADDRESS	DOCUMENT CONTROL NUMBER
Cynthia Heeb Clark	Laboratory PM/ Representative for Laboratory and Analytical Issues	APPL, Inc. (APPL) 908 N. Temperance Avenue Clovis, CA 93611	(559) 275-2175	<a href="mailto:cclark@applinc.com">cclark@applinc.com</a>	NA
TBD	Drilling Subcontractor	TBD	TBD	TBD	NA
TBD	Geophysical Laboratory	TBD	TBD	TBD	NA

**SAP Worksheet #4 – Project Personnel Sign-Off Sheet**  
 (UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters or e-mails will be retained in the project files and are listed in Worksheet #29 as project records.
2. E-mails will be sent to the Navy, Tetra Tech, and subcontractor project personnel whom will be requested to verify by e-mail that they have read the applicable SAP/sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and identified in Worksheet #29.

A copy of the signed Worksheet #4 will be retained in the project files and identified as a project document in Worksheet #29.

NAME	ORGANIZATION/ TITLE/ ROLE	TELEPHONE NUMBER	SIGNATURE/E-MAIL RECEIPT	SAP SECTION REVIEWED	DATE SAP READ
<b>Navy and Regulator Project Team Personnel</b>					
Robert Fisher	NAVFAC SE, RPM/ Manages Project Activities for the Navy	(904) 542-6827	See Worksheet #1 for signature	All	
Gordon Crane	Navy, NCBC Gulfport POC/ Environmental Coordinator	(228) 871-2485		All	
Bob Merrill	MDEQ, RPM/ Provides Regulator Input	(601) 961-5049	Approval was obtained via separate letter	All	

NAME	ORGANIZATION/ TITLE/ ROLE	TELEPHONE NUMBER	SIGNATURE/E-MAIL RECEIPT	SAP SECTION REVIEWED	DATE SAP READ
TBD	USEPA Region 4*, RPM/ Receives Final Document	TBD		TBD	
<b>Tetra Tech Project Team Personnel</b>					
Gregory Roof	Tetra Tech, TOM/ Manages Project Activities	(904) 730-4669 Ext 215	See Worksheet #1 for signature	All	
William Olson	Tetra Tech, FOL/SSO, Lead Geologist/ Manages Field Operation and Site Safety Issues	(850) 385-9866 Ext 1359		All	
Matt Kraus	Tetra Tech, Project Chemist/ Provides Coordination with Laboratories	(412) 921-8729		All	
Kelly Carper	Tetra Tech, QAM/ Manages NAVFAC SE contract QA Program and Implementation	(412) 921-7273	See Worksheet #1 for signature	All	
Joseph Samchuck	Tetra Tech, DVM/ Manages Data Validation	(412) 921-8510		Worksheets #14, #15, #19, #20, #23-28, #30, and #34-37	

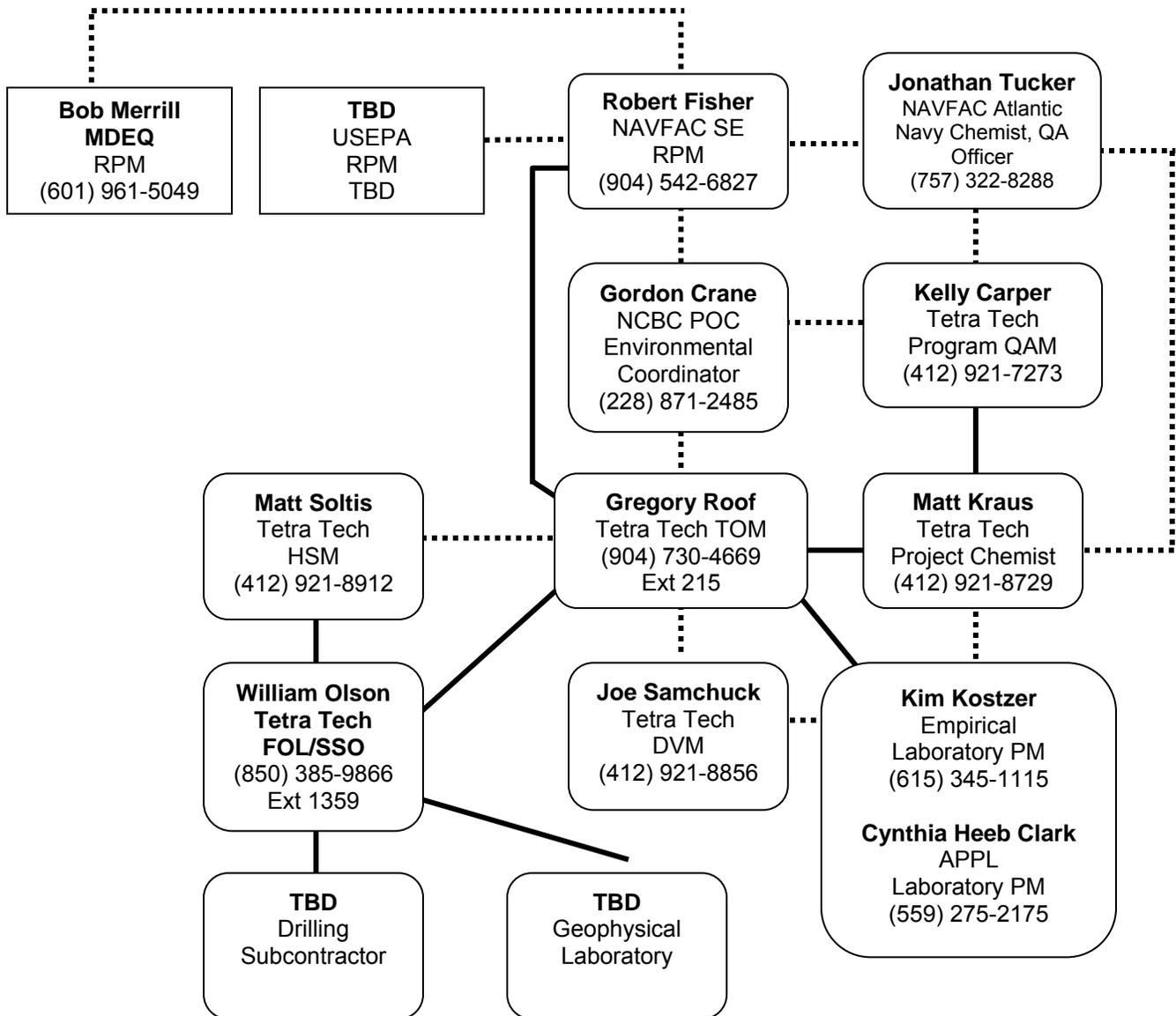
\*USEPA involvement in NCBC Gulfport has been limited to requesting final documents,

NAME	ORGANIZATION/ TITLE/ ROLE	TELEPHONE NUMBER	SIGNATURE/E-MAIL RECEIPT	SAP SECTION REVIEWED	DATE SAP READ
<b>Subcontractor Personnel</b>					
Kim Kostzer	Empirical, Laboratory PM/ Representative for Laboratory and Analytical Issues	(615) 345-1115		Worksheets #6, #10, #11, #15, #19, #23, #24, #25, #26, #27, #28, and #34-#36	
Cynthia Heeb Clark	APPL, Laboratory PM/ Representative for Laboratory and Analytical Issues	(559) 275-2175		Worksheets #6, #10, #11, #15, #19, #23, #24, #25, #26, #27, #28, and #34-#36	
TBD	Drillers for Monitoring Well Installation	TBD		Worksheets #6, #10, #14, and #17	
TBD	Geophysical Laboratory	TBD		Worksheets #6, #10, #14, and #17	

**SAP Worksheet #5 – Project Organizational Chart**  
 (UFP-QAPP Manual Section 2.4.1)

Lines of Authority —————

..... Lines of Communication



**SAP Worksheet #6 – Communication Pathways**  
 (UFP-QAPP Manual Section 2.4.2)

COMMUNICATION DRIVERS	RESPONSIBLE PERSON AFFILIATION	NAME	PHONE NUMBER AND/OR E-MAIL	PROCEDURE
SAP amendments	Tetra Tech FOL/SSO  Tetra Tech TOM  NAVFAC SE RPM	William Olson  Gregory Roof  Robert Fisher	(850) 385-9866 Ext 1359 (904) 730-4669 Ext 215 (904)542-6827	The Tetra Tech FOL will verbally inform the Tetra Tech TOM within 24 hours of realizing a need for an amendment.  The Tetra Tech TOM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change.  SAP amendments will be submitted by the Tetra Tech TOM to the Navy RPM for review and approval.  The Tetra Tech TOM will send scope changes to the Project Team via e-mail within 1 business day.
Changes in schedule	Tetra Tech TOM  NAVFAC SE RPM  NCBC Gulfport POC	Gregory Roof  Robert Fisher  Gordon Crane	(904) 730-4669 Ext 215 (904) 542-6827  (228) 871-2485	The Tetra Tech TOM will verbally inform the NAVFAC SE RPM and the NCBC Gulfport POC on the day that schedule change is known and document via schedule impact letter within 1 business day of when impact is realized.
Issues in the field that lead to changes in the scope of work	Tetra Tech FOL/SSO  Tetra Tech TOM	William Olson  Gregory Roof	(850) 385-9866 Ext 1359  (904) 730-4669 Ext 215	The Tetra Tech FOL will verbally inform the Tetra Tech TOM on the day that the issue is discovered.  The Tetra Tech TOM will inform the NAVFAC SE RPM and the NCBC Gulfport POC (verbally or via e-mail) within 1 business day of discovery.  The NAVFAC SE RPM will issue scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed.  The Tetra Tech TOM will document the change via an FTMR form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form.

COMMUNICATION DRIVERS	RESPONSIBLE PERSON AFFILIATION	NAME	PHONE NUMBER AND/OR E-MAIL	PROCEDURE
Recommendation to stop work and initiate work upon corrective action (CA)	Tetra Tech FOL/SSO	William Olson	(850) 385-9866 Ext 1359	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the NCBC Gulfport POC, and the identified Project Team members within one hour (verbally or by e-mail).  If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
	Tetra Tech TOM	Gregory Roof	(904) 730-4669 Ext 215	
	Tetra Tech QAM	Kelly Carper	(412) 921-7273	
	NAVFAC SE RPM	Robert Fisher	(904) 542-6827	
	NCBC Gulfport POC	Gordon Crane	(228) 871-2485	
CA for field program	Tetra Tech QAM	Kelly Carper	(412) 921-7273	The Tetra Tech QAM will notify the Tetra Tech TOM verbally or by e-mail within 1 business day that the CA has been completed.  The Tetra Tech TOM will then notify the Navy RPM within 1 business day.
	Tetra Tech TOM	Gregory Roof	(904) 730-4669 Ext 215	
Field data quality issues	Tetra Tech FOL/SSO	William Olson	(850) 385-9866 Ext 1359	The Tetra Tech FOL will inform the Tetra Tech TOM verbally or by e-mail on the same day that a field data quality issue is discovered.
	Tetra Tech TOM	Gregory Roof	(904) 730-4669 Ext 215	
Analytical data quality issues	Empirical PM	Kim Kostzer	(615) 345-1115	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered.  The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech TOM within 1 business day.
	APPL PM	Cynthia Heeb Clark	(559) 275-2175	
	Project Chemist	Matt Kraus	(412) 921-8729	

**SAP Worksheet #7 – Personnel Responsibilities and Qualifications Table**  
 (UFP-QAPP Manual Section 2.4.3)

The personnel from Tetra Tech and the analytical laboratories responsible for implementing the SAP are identified in the following table. Resumes are available upon request.

NAME	TITLE/ROLE	ORGANIZATIONAL AFFILIATION	RESPONSIBILITIES
Gregory Roof	TOM	Tetra Tech	<p>Oversees project, manages financial, schedule, and technical day-to-day activities of the project, including the following:</p> <ul style="list-style-type: none"> <li>• Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations.</li> <li>• Functions as the primary Tetra Tech interface with the NAVFAC SE RPM, Base personnel, Tetra Tech field and office personnel, and laboratory POCs.</li> <li>• Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.</li> <li>• Monitors and evaluates all Tetra Tech subcontractor performance.</li> <li>• Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data validation, data interpretation, and report preparation).</li> <li>• Coordinates and oversees maintenance of all Tetra Tech project records.</li> <li>• Coordinates and oversees review of Tetra Tech project deliverables.</li> <li>• Prepares and issues Tetra Tech deliverables to the Navy and Project Team.</li> </ul>

NAME	TITLE/ROLE	ORGANIZATIONAL AFFILIATION	RESPONSIBILITIES
Kelly Carper	QAM	Tetra Tech	<p>Approves SAP and ensures that quality aspects of the CLEAN program are implemented, including the following:</p> <ul style="list-style-type: none"> <li>• Develops, maintains, and monitors QA policies and procedures.</li> <li>• Provides training to Tetra Tech staff in QA/Quality Control (QC) policies and procedures.</li> <li>• Conducts management and technical audits to monitor compliance with environmental regulations, contractual requirements, Quality Assurance Project Plan (QAPP) requirements, and corporate policies and procedures.</li> <li>• Audits project records.</li> <li>• Monitors subcontractor quality controls and records.</li> <li>• Assists in the development of CA plans and ensuring correction of non-conformances reported in internal or external audits.</li> <li>• Ensures that this SAP meets Tetra Tech, Navy, USEPA and MDEQ requirements.</li> <li>• Prepares QA reports for management.</li> </ul>

NAME	TITLE/ROLE	ORGANIZATIONAL AFFILIATION	RESPONSIBILITIES
William Olson	FOL/SSO	Tetra Tech	<p>Supervises, coordinates, and performs field sampling activities, including the following:</p> <ul style="list-style-type: none"> <li>• Ensures that all health and safety requirements applicable to the field work are implemented.</li> <li>• Functions as the on-site communications link between field staff members, NAVFAC SE RPM, NCBC Gulfport personnel, and the Tetra Tech TOM.</li> <li>• Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples.</li> <li>• Oversees the mobilization and demobilization of all field equipment and subcontractors.</li> <li>• Coordinates and manages the field technical staff.</li> <li>• Adheres to the work schedules provided by the Tetra Tech TOM.</li> <li>• Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping.</li> <li>• Initiates FTMR forms (if necessary).</li> <li>• Identifies and resolves problems in the field, resolves difficulties via consultation with the Tetra Tech TOM, Navy RPM, and NCBC Gulfport personnel, implements and documents CAs related to field work, and serves as communication link between the field team and project management.</li> <li>• As the SSO, is responsible for training and monitoring site conditions. The SSO reports to the Company Health and Safety Officer and to the Tetra Tech TOM. Details of the SSO's responsibilities are presented in the Health and Safety Plan (HASP).</li> </ul>

NAME	TITLE/ROLE	ORGANIZATIONAL AFFILIATION	RESPONSIBILITIES
Matt Kraus	Project Chemist	Tetra Tech	<p>Prepares laboratory scopes of work, coordinates analyses with laboratory chemists, ensures that the laboratory scope of work is followed, and communicates with Tetra Tech staff. Performs data quality reviews.</p> <ul style="list-style-type: none"> <li>• Ensures that the project meets objectives from the standpoint of laboratory performance.</li> <li>• Provides technical advice to the Project Team on matters of data quality and project chemistry.</li> <li>• Monitors and evaluates subcontractor laboratory performance.</li> <li>• Ensures timely resolution of laboratory-related technical, quality, or other issues affecting project goals.</li> <li>• Functions as the primary interface between the subcontracted laboratories and the Tetra Tech TOM.</li> <li>• Coordinates and oversees work performed by the subcontracted laboratories.</li> <li>• Oversees the completion of Tetra Tech data validation.</li> <li>• Coordinates and oversees review of laboratory deliverables.</li> <li>• Recommends appropriate laboratory CAs.</li> </ul>
Joseph Samchuck	DVM	Tetra Tech	<p>Provides QA of data validation deliverables, including the following:</p> <ul style="list-style-type: none"> <li>• Oversees data validation activities.</li> <li>• Serves as communication link between Tetra Tech and laboratories on data validation and electronic data positing activities.</li> <li>• Establishes Tetra Tech data validation protocols in support of projects.</li> </ul>
Matt Soltis	HSM	Tetra Tech	<p>Oversees CLEAN Program Health and Safety Program.</p> <ul style="list-style-type: none"> <li>• Provides technical advice to the Tetra Tech TOM on matters of health and safety.</li> <li>• Oversees the development and review of the HASP.</li> <li>• Conducts health and safety audits and prepares health and safety reports for management.</li> </ul>

NAME	TITLE/ROLE	ORGANIZATIONAL AFFILIATION	RESPONSIBILITIES
Kim Kostzer  Cynthia Heeb Clark	Laboratory PM  Laboratory PM	Empirical  APPL	Interfaces directly with the Tetra Tech Project Chemist, Tetra Tech TOM, and Tetra Tech QAM. <ul style="list-style-type: none"> <li>• Ensures that methods and project-specific requirements are properly communicated and understood by laboratory personnel.</li> <li>• Ensures that all laboratory resources are available on an as required basis.</li> <li>• Ensures compliance with analytical and project QA requirements.</li> <li>• Reviews data packages for completeness, clarity, and compliance with project requirements.</li> <li>• Informs the Tetra Tech TOM of project status and any sample receipt or analytical problems.</li> <li>• Oversees the preparation of and approves final analytical reports before submittal to Tetra Tech.</li> </ul>
TBD	Drillers	Subcontractor	Install monitoring wells.

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL will be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

**SAP Worksheet #8 – Special Personnel Training Requirements Table**  
(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker performing sampling of hazardous materials will be required to have completed a 40-hour course (and annual 8-hour refresher) in Health and Safety Training as described under Occupational Safety and Health Administration 29 Code of Federal Regulations 1910.120(b)(4). Safety requirements are addressed in greater detail in the Tetra Tech site-specific HASP (Tetra Tech, 2009) that was previously submitted as a separate document to the Navy.

**SAP Worksheet #9 – Project Scoping Session Participants Sheet**  
 (UFP-QAPP Manual Section 2.5.1)

Project Name: NCBC Gulfport		Site Name: Site 2 – World War II Landfill			
Projected Date(s) of Sampling: <u>September 2010</u>		Site Location: NCBC Gulfport, Mississippi			
Project Manager: Greg Roof					
<b>Date of Session:</b> May 13, 2009					
<b>Scoping Session Purpose:</b> DQO Section in Partnering Meeting					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Robert Fisher	NAVFAC SE RPM	NAVFAC SE	(904) 542-6827	<a href="mailto:robert.r.fisher@navy.mil">robert.r.fisher@navy.mil</a>	NAVFAC SE RPM
Gordon Crane	NCBC POC	NCBC Gulfport	(228) 871-2485	<a href="mailto:gordon.crane@navy.mil">gordon.crane@navy.mil</a>	NCBC POC
Bob Merrill	MDEQ RPM	MDEQ	(601) 961-5049	<a href="mailto:bob_merrill@deq.state.ms.us">bob_merrill@deq.state.ms.us</a>	RPM
Nancy Rouse	Facilitator	The Management Edge	(760) 470-0751	<a href="mailto:nvrouse@aol.com">nvrouse@aol.com</a>	Facilitator
Jacqueline Strobl	Project Assistant	Tetra Tech	(850) 385-9899 Ext 1351	<a href="mailto:jacqueline.strobl@tetrattech.com">jacqueline.strobl@tetrattech.com</a>	Scribe
Jon Overholtzer	PM	CH2M Hill	(678) 530-4262	<a href="mailto:jon.overholtzer@ch2m.com">jon.overholtzer@ch2m.com</a>	Remedial Action Contract PM
Greg Roof	TOM	Tetra Tech	(904) 730-4669 Ext 215	<a href="mailto:gregory.roof@tetrattech.com">gregory.roof@tetrattech.com</a>	TOM
Peggy Churchill	DQO Facilitator	Tetra Tech	(321) 636-6470	<a href="mailto:peggy.churchill@tetrattech.com">peggy.churchill@tetrattech.com</a>	QAPP/DQO Facilitator
Helen Lockard	Tier II Link	NAVFAC SE	(904) 542-6858	<a href="mailto:helen.lockard@navy.mil">helen.lockard@navy.mil</a>	Tier II Link

<b>Consensus/Decisions Items</b> (The DQO meeting minutes, which are part of the Partnering Minutes, are included in Appendix A.)	
1	Information from the groundwater sampling collected in 1985 should not be used because the monitoring wells were incorrectly placed upgradient.
2	Containment, as part of a Presumptive Remedy, will be considered as a remedial action. Therefore, this remedial investigation (RI) should be focused on supporting or complementing the Presumptive Remedy approach.
3	Michigan Department of Environmental Quality "Sampling Strategies and Statistics Training Materials for Part 201 Cleanup Criteria (S3TM)" is MDEQ's preferred method for determining the sampling strategy. This guidance document will be taken into consideration for the sampling design.
4	Selective analysis for dioxins will be limited to surface soil, groundwater, and sediment in select locations.

**SAP Worksheet #10 – Conceptual Site Model**  
**(UFP-QAPP Manual Section 2.5.2)**

**10.1 SITE BACKGROUND**

NCBC Gulfport is located in the western part of Gulfport, Mississippi, in Harrison County, in the southeastern corner of the state approximately 2 miles north of the Gulf of Mexico. The property for the installation was acquired in April 1942 and occupies approximately 1,100 acres. Site 2 is located on the eastern side of Colby Avenue, between 8<sup>th</sup> and 11<sup>th</sup> Streets, within the boundaries of NCBC Gulfport (see Figure 2).

Site 2, known as the World War II Landfill, was reportedly operational from 1942 to 1948 during which time it was the primary disposal area for general municipal-type refuse generated at NCBC Gulfport. The disposal operation consisted of burning combustible materials in a structure formerly located at the northern end of the site, then pushing the remaining non-combustible material and ash to the southern end of the site for burial. Waste materials were buried more than 8 feet deep in trenches that typically contained standing water and, thus, brought the waste materials into direct contact with surficial ground water. Upon disposal in the trench, the waste materials were covered with soil.

Available records indicate that landfill activities at Site 2 ceased in 1948. Land use between the reported closure of the landfill in 1948 and the next documented use in 1984 is unknown. The land was undeveloped at the time of the 1984 Site Inspection performed by Envirodyne Engineers, Inc. and remained undeveloped until the current golf course fairway was constructed in late 1998.

Site 2 is the fifth landfill at NCBC Gulfport that will be undergoing an RI. Site 1, the Disaster Recovery Area; Site 3, the Northwest Landfill; Site 4, the Golf Course Landfill; and Site 5, the Heavy Equipment Training Area Landfill, are the other four former landfills sites that have been investigated. Site 7, the Rubble Disposal Area, operated as a landfill from 1978 until 1984 and is located just north of Site 2 and is currently also used as a fairway for the Pine Bayou Golf Course; however, no RIs have been conducted at Site 7 to date. Data gathered during the RI phase from Sites 1, 3, 4, and 5 showed the sites could be managed using the Presumptive Remedy approach. Presumptive Remedies are preferred technologies for common categories of sites based on historical patterns of remedy selection and the USEPA's scientific and engineering evaluation of performance data on technology implementation. The objective of the Presumptive Remedy for CERCLA Municipal Landfill Sites is to use the program's past experience to streamline site investigation and selection of remedial alternatives (USEPA, 1993). Therefore, the Project Team has decided that based on the history and investigations at nearby similar landfills and according to USEPA guidance, the Presumptive Remedy for containment at municipal type landfills will be applied at Site 2.

## **10.2 SUMMARY OF PREVIOUS INVESTIGATIONS**

### **10.2.1 Initial Assessment Study**

During the Initial Assessment Study (IAS), sites at NCBC Gulfport that were potential threats to human health and the environment were identified (Envirodyne Engineers, Inc., 1985). The IAS included a records search, on-site survey, site ranking, and an outline for a subsequent confirmation study. The IAS stated that at Site 2 there is potential for contaminant migration due to portions of the waste being in direct contact with surficial groundwater. Based on this information, a confirmation study was recommended in the IAS.

### **10.2.2 Verification Report**

The Verification Report (applicable sections included in Appendix B) was conducted by Harding Lawson Associates (HLA) in 1988 and included the results of site reconnaissance, a geophysical survey, and an investigation of soil, surface water, groundwater, and sediment.

During the geophysical survey, very low frequency (VLF) electromagnetic data indicating variations in soil conductivity and magnetometer data indicating variations in the total magnetic field associated with magnetic objects were collected. The grid spacing for this survey was 50 feet. Almost 40 percent of Site 2 exhibited VLF values greater than the background value, suggesting that native soil had been disturbed by excavation and disposal activities. The magnetometer data identified an anomalous area occupying one-third of the western one-half of Site 2 and additional magnetic anomalies in the north central portion of the site. However, this geophysical study is considered only as reference because the spacing between the grids was determined to be too wide.

One surface water sample (SW2-1) and one sediment sample (SD2-1) were collected during the Verification Study. These samples were collected from the drainage ditch near the southeastern side of the intersection of Colby Avenue and 8<sup>th</sup> street (see Plate 6 in Appendix B. For proper orientation, note the direction of the North arrow). The samples were analyzed for selected metals (cadmium, chromium, and lead), oil and grease, total organic carbon (TOC), total organic halides, and chemical oxygen demand. Low levels of chromium and lead were detected below regulatory levels in the sediment sample. Other metal concentrations were less than the laboratory detection limits.

Three monitoring wells were installed and sampled in March 1987 as part of the Verification Study. These wells (GPT-2-1, GPT-2-2, and GPT-2-3) are depicted in Plate 6 in Appendix B. The location of GPT-2-1 and GPT-2-2 are on the southern border of Site 2 and GPT-2-3 is located on the northern border of Site 7. The groundwater samples were analyzed for volatile organic compounds (VOCs),

base/neutral/acid (BNA) extractable organics, and selected metals (cadmium, chromium, and lead). Analytical results showed very low levels of chlorinated organic contaminants were detected below regulatory levels only in monitoring well GPT-2-3 (HLA, 1988).

### **10.2.3 Basewide Groundwater, Surface Water, and Sediment Investigation**

A basewide sampling event was conducted in 1994 to investigate the groundwater conditions of 6 sites, including Site 2, at NCBC Gulfport. Three monitoring wells (GPT-2-1, GPT-2-2, and GPT-2-3) were redeveloped and sampled in December 1994 for VOCs and semivolatile organic compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), metals, herbicides, dioxins and furans, and total petroleum hydrocarbons (TPH) (ABB, 1995). As mentioned in Section 10.2.2, wells GPT-2-1 and GPT-2-2 are located on the southern border of Site 2 along 8<sup>th</sup> Street and GPT-2-1 no longer exists. GPT-2-3 was located on the north side of Site 7 south of 15<sup>th</sup> street (See Figure 6A).

The results of the 1994 basewide sampling event indicated a detection of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) within monitoring well GPT 2-3 at 5.5 picograms per liter (pg/L). Monitoring well GPT 2-3 is located north of the Site 2 north-east Sampling Grid Boundary by approximately 475 feet and hydraulically side gradient of Site 2 (see Figure 6A). Organic compounds and herbicides were not detected in any of the groundwater samples. Metals were detected in all of the groundwater samples collected in this investigation. The recommendations included further investigation of Sites 4 and 5 and re-sampling at Site 7 due to the TCDD detection.

### **10.2.4 Groundwater Monitoring Investigation**

An additional groundwater investigation was conducted at NCBC Gulfport to determine the extent of dioxin and dioxin-related chemicals at Sites 4, 5, and 8. Phase II of this investigation included the installation and sampling of permanent monitoring wells downgradient of Sites 1, 2, 3, and 7 based on surficial aquifer flow directions. None of the newly installed downgradient monitoring wells at Sites 1, 2, and 3 contained measured concentrations of dioxins.

One monitoring well at Site 7 (GPT-7-1) was installed on February 16, 1999, and sampled on February 23, 1999. The groundwater sample was reported to contain 51.6 pg/L dioxin with an estimated 25 pg/L attributed to TCDD, the main herbicide orange (HO) dioxin congener (Harding Lawson Associates, 1999). The 51.6 pg/L dioxin concentration exceeded the current Maximum Contaminant Level (MCL) of 30.0 parts per quadrillion. Well GPT-7-1 is located approximately 100 feet north of the Sampling Grid Boundary for Site 2. Recommendations resulting from the groundwater study include no further study of groundwater at Sites 1, 2, 3, and 8; and an investigation for dioxin in groundwater at

Site 7; Figure 6A depicts the location of monitoring well GPT-7-1 in relation to Site 2 Investigation Boundary.

### **10.3 CONSTRUCTION ACTIVITIES**

Site 2 remained undeveloped after disposal activities were discontinued around 1948. The IAS reported that the site was undeveloped. In 1994, the Navy was notified of a sheen that was seen in the nearby ditch (a copy of such notification is included in Appendix B); however, it is unknown if this was investigated further. Afterward, a fact sheet distributed to the community in 1996 described the site as planted pine forest with dense underbrush (also included in Appendix B). Later, in 1998, the golf course was expanded and the tee and fairway for the 12<sup>th</sup> and 15<sup>th</sup> holes were constructed on the site. According to data collected at nearby sites (Sites 1, 3, 4, and 5), the golf course was constructed by adding anywhere from 6 inches to over 2 feet of top soil.

### **10.4 CONCEPTUAL SITE MODEL**

For a standard approach to documenting Conceptual Site Model (CSM) information, MDEQ recommends the use of the schematic Baseline Site Conceptual Exposure Model (BSCEM) to evaluate the sources, transport mechanisms, exposure pathways, and receptors. Within the BSCEM, surface soil is defined as less than 6 feet from land surface. For purposes of this investigation, surface soil is not anticipated to be investigated; however, subsurface soil beneath the fill that was put in place when the golf course was constructed may be investigated. The BSCEM is included as Figure 3 and a CSM schematic is presented as Figure 4.

#### **10.4.1 Sources and Potential Contaminants**

The primary source of contamination at Site 2 is the refuse that was disposed when the site was used as an active landfill. The majority of the waste disposed at the site was general refuse and inert material such as paper, cardboard, wood, and household garbage. Liquid waste such as paints, paint thinners, solvents, oils, and fuels were also reportedly disposed at the site (incinerated or buried). There is no documentation indicating the exact volume of waste that was disposed at the site. Paints used at NCBC Gulfport during the time Site 2 was operational typically contained cadmium, lead, and chromium. These metals, as well as products of incomplete combustion and dioxins/furans formed during combustion, may exist at the site.

As noted in the previous paragraph, the majority of the waste disposed at Site 2 was general refuse. However, dioxins were detected in the groundwater associated with Site 7, which is adjacent to Site 2. In addition HO was held at other sites on NCBC Gulfport and dioxins, in particular TCDD, are a byproduct

contaminant of the manufacturing process associated with HO (Agent Orange/Dioxin Committee, 2002). Therefore, dioxins as they may be associated with HO and dioxins/furans, and as they may be associated with products of incomplete combustion are potential Site 2 contaminants in addition to the chemical constituents commonly associated with general refuse (e.g. metals, VOCs, SVOCs, pesticides, herbicides, and PCBs)

#### **10.4.2 Contamination Migration Pathways**

Because waste material may be present in the subsurface, subsurface soil may be contaminated and groundwater that encounters the waste may become contaminated. As the groundwater migrates through the site, downgradient subsurface soil and groundwater may also be impacted. The site is currently covered with fill that ranges in thickness from 6 inches to over 2 feet because of golf course construction. Site 2 surface water flows toward the pond on the eastern side of the site and toward the ditch to the west. This water is then conveyed toward storm water Outfalls 1 and 3 on the northern end of the site as shown on Figure 5. Groundwater flow is most likely towards the pond; however, there is a potential groundwater divide that causes groundwater to flow to the west as well. In this case, it may recharge the western ditch. Groundwater elevation data will be collected as part of this investigation and analyzed to determine the direction of groundwater flow.

#### **10.4.3 Receptors and Exposure Pathways**

Potential human receptors at Site 2 include people currently employed at the site, trespassers, maintenance workers, and recreational site users who could potentially interact with contaminated media. There is no future land use planned for the site other than its current use as a golf course. Current and future ecological receptors include the flora (predominantly grassland species) and fauna (earthworms, insect larvae, and other soil invertebrates, herbivorous birds, vermivorous birds, and mammals) present at the site. Additional information regarding the human health and ecological risk assessments can be found in Appendix C. The potential exposure pathways include the ingestion and dermal exposure to groundwater, surface water, and sediment; ingestion and dermal exposure of subsurface soil; and inhalation of subsurface soil dust or vapors.

For purposes of completeness, risk assessments typically evaluate the major land use scenarios hypothetically possible for a site. While a site may never be used for residential purposes, the risk managers must understand the risks associated with such a land use in order to select the appropriate remedy for the site that may include land use controls (LUCs) in addition to the containment presumptive remedy.

## **SAP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements** **(UFP-QAPP Manual Section 2.6.1)**

### **11.1 PROBLEM STATEMENT**

Historic activities at Site 2 included disposal, incineration, and burial of municipal-type waste. In 1998, the golf course fairway and tee boxes were constructed on the surface of the former landfill. This recent construction may limit infiltration into the subsurface, thereby minimizing subsurface impacts and potential contaminant migration. Because of these site characteristics, the Site 2 landfill is appropriate for the application of the Presumptive Remedy for CERCLA Municipal Landfill Sites (USEPA, 1993). A streamlined RI must be conducted to determine if contamination is migrating from the site and if any unacceptable human or ecological risk from exposure to contaminated site media exists. Data gathered from this investigation will also be used to support landfill cover assessment and design, which will be presented in the Feasibility Study (FS).

### **11.2 INFORMATION INPUTS**

In order to meet the study goals of the RI, the physical and chemical data to be collected at Site 2 are described below. The Field Documentation Standard Operating Procedure (SOP), included in Appendix D, requires the field crew to keep a log regarding observations made while collecting subsurface soil samples. These observations will assist to understand the composition of the landfill.

#### **11.2.1 Geophysical Survey**

Initial field investigation activities will include an electromagnetic geophysical survey of the site in order to determine the physical extent of surficial and buried solid waste material. Because environmental media may be contaminated in and near such waste, these measurements will help to define the majority of contamination and identify potential contamination sources. Identification of geophysical anomalies will assist with the selection of sample locations. Soil and groundwater sample locations will be adjusted in the field if necessary to ensure that a sample is collected from potential waste disposal areas identified as anomalies.

#### **11.2.2 Soil Gas Survey**

Passive soil gas samplers will be used to screen for VOC hot spots in the shallow subsurface. The passive soil gas samplers provide semi-quantitative data for the occurrence of VOCs in the vadose zone and shallow groundwater. The soil gas survey results will assist with determining soil and groundwater sample locations.

### **11.2.3 Landfill Gas Survey**

A landfill gas survey to assess the methane gas production of the former landfill is planned. To determine if a gas collection/venting system should be included as a component of the final cover, field monitoring will be conducted to evaluate the presence, concentrations, and types of gasses present.

Gas composition will be evaluated in the field by collecting and analyzing samples from temporary probe points using a landfill gas analyzer (for percent of methane, carbon monoxide, and oxygen) and a multi-gas meter (photoionization detector [PID]) (for oxygen, carbon dioxide, and explosive limit). Gas samples will be collected at 41 locations on the landfill at known disposal cell locations. Gas samples will also be collected from gas migration and potential receptor locations and the edges of the current cover.

### **11.2.4 PID Subsurface Soil Screening**

PID subsurface soil screening data will be used to identify potential source locations and select the subsurface soil interval from the soil borings with the highest concentration of volatile contaminants for sampling purposes.

### **11.2.5 Field Investigation Parameters**

Water table level, groundwater dissolved oxygen, conductivity, pH levels, temperature, turbidity, and oxidation-reduction potential data will assist with site characterization and, when combined with chemical soil and groundwater data, will assist with understanding the nature and extent of site contamination. The groundwater measurements will be used to determine when groundwater samples are representative of the groundwater from the aquifer being investigated.

### **11.2.6 Chemical Analysis**

Subsurface soil, sediment, surface water, and groundwater chemical data will be used to determine the nature and extent of VOCs, SVOCs, inorganics (metals and cyanide), pesticides, herbicides, and PCBs. The field event, which will be divided into four events as described in Worksheet #14, will accommodate a preliminary round of sampling based on the results of the soil gas and geophysical survey and a later sampling round based on the preliminary sampling. Additionally, soil, groundwater, and sediment samples will be analyzed for dioxins and furans as agreed during the DQO meeting and previous experiences. The list of all chemical analytical groups and individual target analytes within each group is presented in Worksheet #15. The sampling methods are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19. The selected target analytes represent those analytes that are potentially associated with historical site operations as identified in the CSM in Section 10.3.

### 11.2.7 Project Action Limits

The RI requires chemical data that can be used define the extent of potential contamination and to conduct an ecological and human health risk assessment. Chemical concentrations will be compared to conservative screening values, which are the Project Action Limits (PALs). To conduct comparisons of site data to screening values, the selected laboratory must be able to achieve Limits of Quantitation (LOQs) that are low enough to measure constituent concentrations below the screening values. For this investigation the screening values, which are also known as the PALs, for all media are included in Worksheet #15.

Analytical data reported by the laboratory use the following reporting conventions: All results below the Detection Limit (DL) will be considered non-detects; positive results reported at concentrations between the DL and LOQ will be reported with a "J" qualifier; and analytes not found (not detected) in a sample will be reported as the Limit of Detection (LOD) with a "U" qualifier.

For risk assessment purposes, in the event that an analyte concentration exceeds a PAL but is less than or equal to an established background concentration, the analyte will not be considered a contaminant of potential concern. For delineation purposes, if a background concentration for a particular analyte is greater than the PAL for that analyte, the background concentration will replace the PAL.

Additional screening levels for other USEPA Regions have been included in Worksheet #15 to ensure laboratory sensitivity is sufficient. PALs were selected by choosing the lowest value among Ecological Soil Screening Levels (Eco-SSL) for plant, invertebrate, mammalian and avian values were selected as the ecological screening value (ESV). Eco-SSLs were used preferentially as soil screening values; however, Eco-SSLs are currently available for only a few analytes. USEPA Region 4 ESVs (USEPA, 2001) were used as screening values for analytes that do not have an Eco-SSL. The term "Soil ESV" is used for brevity in this SAP to refer to either the Eco-SSL or the Region 4 Soil ESV. Additional information regarding the ecological risk methodology proposed for this RI is included in Appendix C.

Several target analytes have PALs that fall between the LOD and the LOQ. J-flagged data will be accepted to achieve project goals; however, greater scrutiny will be applied in these cases. Additionally, the inability to quantify select analytes to PAL levels with confidence will be addressed in the risk assessment uncertainty analysis.

In cases where the laboratory LODs are greater than the PALs, as per the Partnering Team meeting held on May 12 and 13, 2009, and consistent with the USEPA *Risk Assessment Guidance for Superfund*, Part A (USEPA, 1989), if the analyte is not detected, the LOD will be reported and "U" qualified. An

evaluation of these analytes will be also presented in the uncertainty section of the risk assessment in the RI Report.

### 11.3 STUDY AREA BOUNDARIES

Site 2 is spatially bounded by Colby Avenue to the west, 8<sup>th</sup> Street to the south, and wooded and vacant land to the north and east. A pond is located on the eastern side the site. The horizontal boundary is currently based on aerial photographs and historical geophysical results. The final horizontal boundary will be determined by the results of a 20-foot grid geophysical study to be performed in the initial stages of the RI. Geophysical data representative of any buried waste or disturbances in the subsurface will be used for determining the landfill boundaries.

The vertical boundary for soil extends to a depth of 40 feet below land surface (bls). During the investigation, data that is representative of three populations of interest will be collected from three different soil intervals, which include soil above the surficial aquifer, soil between the surficial aquifer and intermediate aquifer, and the soil between the intermediate aquifer and the deep aquifer. These populations represent soil that may be contaminated due to activities or disposal on the surface or near the surface (when the landfill was in use), soil that may be impacted by potentially contaminated groundwater in the surficial aquifer, and soil that may be impacted by potentially contaminated intermediate groundwater respectively. Once a boring has been installed, soil gas data and PID readings will be collected from the three soil intervals of interest. If no volatile compounds are detected during field screening, no samples from the intervals below the surficial aquifer will be collected and sent to the laboratory for analysis. Data will only be collected from two populations that are representative of any contamination that may have migrated from the surface, which include the first 0- to 6-inch interval below the fill material used to construct the golf course and the 0- to 6-inch interval just above the water table.

The vertical boundary for groundwater includes the depth to the deeper aquifer, which is approximately 40 feet bls. The groundwater populations of interest include the shallow or surficial aquifer, intermediate aquifer, and deep aquifer. Representative samples from the populations of interest will be collected and used for decision making.

The sediment interval of interest is 0- to 6-inches bls, which defines the vertical boundary for this media. The population of interest for sediments includes any sediment along the western shoreline of the pond that may be impacted by contaminated groundwater that potentially migrates from the site and recharges the pond. Surface water data collected from the pond is representative surface water that may be contaminated due to migration of contaminated site media.

## **11.4 ANALYTIC APPROACH**

The analytic approach for the RI includes decision rules related to characterizing the site to determine if the Presumptive Remedy is appropriate, delineate potential contamination, and assess potential risk. Additionally, QA data to be collected is described in Worksheets #20 and #28.

Due to limitations in analytical sensitivity, some analytes cannot be detected at their PAL. These analytes are shown as bolded and shaded on worksheet #15. If these analytes are reported as non-detected with U-qualifiers, they will be treated as results below the project action limit for decision making.

### **11.4.1 Site Characterization**

In order to determine if the Presumptive Remedy is appropriate for the site, the Project Team has agreed that the following criteria been met:

- Risks are low-level, except for hotspots.
- Waste types are generally household, commercial, non-hazardous sludge, and industrial solid wastes.
- Lesser quantities of hazardous wastes are present as compared to municipal-type wastes, if any.
- No hazard military-specific wastes (such as unexploded ordnance, radioactive waste, or biological/chemical warfare agents).

Following the investigation of the site, the Project Team will evaluate the results. If any of the above criteria are not met, the Project Team will evaluate in the FS other remedial alternatives that are more appropriate for the site. If the above criteria are met, the Project Team will apply the Presumptive Remedy at the site.

### **11.4.2 Containment Analytic Approach**

The perimeter of the landfill, as defined by the geophysical survey, will be investigated by comparing media concentrations to the PALs. If the data are sufficient to define the extent of contamination at the site, data collection will cease. If the data are not sufficient to delineate the extent of contamination, then data collection will continue to step out field screening samples until the extent of contamination is known.

Data will be gathered in phases as presented in Worksheet #17. If chemical concentrations in subsurface soil and/or groundwater exceed PALs, additional data will be collected for delineation. If chemical concentrations in subsurface soil and/or groundwater do not exceed PALs, then no additional data will be collected for delineation.

### **11.4.3 Risk Assessment Analytic Approach**

In order to determine whether ecological and human health risks are acceptable under current site conditions a cancer risk of  $10^{-6}$  and below a hazard quotient of 1 for non-cancer risk will be used. If preliminary risks are shown to be unacceptable, the Project Team will determine if the Containment Presumptive Remedy will adequately mitigate the unacceptable risk. If not, additions to the containment remedy, such as LUCs, will be presented and evaluated in the FS.

### **11.5 PERFORMANCE OR ACCEPTANCE CRITERIA**

Because the biased sampling locations were strategically selected to locate potential contamination and ensure that any landfill-related contaminants are contained within the landfill boundary, probability limits for false positive and false negative decision errors were not established. Simple comparisons of measured concentrations to action levels are being used. The Project Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have action levels that are less than the detection limits (DLs) to ensure that contaminants are likely to have been detected, if present. If all data have been collected as planned and no data points are missing or rejected for quality reasons, the sampling event completeness will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether a claim of having obtained project objectives is reasonable. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented. All stakeholders will be involved in rendering the final conclusion regarding adequacy of the data.

### **11.6 DATA COLLECTION PLAN**

The data collection plan and the sample design rationale for Site 2 are included in Worksheet #17.

**SAP Worksheet #12 – Measurement Performance Criteria Table Field QC Sample – All Fractions**  
 (UFP-QAPP Manual Section 2.6.2)

QC SAMPLE	ANALYTICAL GROUP	FREQUENCY	DATA QUALITY INDICATORS (DQIs)	MEASUREMENT PERFORMANCE CRITERIA (MPC)	QC SAMPLE ASSESSES ERROR FOR SAMPLING (S), ANALYTICAL (A) OR BOTH (S&A)
Field Blank	All Fractions	One per source water.	Accuracy/Bias/Contamination	No analytes > ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment <sup>1,2</sup> .	Accuracy/Bias/Contamination	No analytes > ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Trip Blanks	VOCs	One per cooler containing VOC samples.	Accuracy/Bias/Contamination	No analytes > ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Field Duplicate	All Fractions	One per 10 field samples collected.	Precision	Values > 5X LOQ: Relative Percent Difference (RPD) ≤ 30% <sup>3,4</sup> (aqueous); ≤ 50% <sup>3,4</sup> (solid).	S
Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature between 2 and 6 degrees Celsius (°C) (4 ± 2 °C).	S

Notes:

- 1 Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
- 2 A filter rinsate blank will be collected if filtered samples are collected (i.e., dissolved iron and manganese).
- 3 If duplicate values for non-metals are < 5x QL, the absolute difference should be < 2x LOQ.
- 4 If duplicate values for metals are < 5x LOQ, the absolute difference should be < 4x LOQ.

**SAP Worksheet #13 – Secondary Data Criteria and Limitations Table**  
 (UFP-QAPP Manual Section 2.7)

SECONDARY DATA	DATA SOURCE (originating organization, report title and date)	DATA GENERATOR(S) (originating organization, data types, data generation / collection dates)	HOW DATA WILL BE USED	LIMITATIONS ON DATA USE
IAS	Originating Organization: Envirodyne Engineers, Inc.  Report Title: <i>Initial Assessment Study for Naval Construction Battalion Center</i>  Date: July 1, 1985	Originating Organization: Envirodyne Engineers, Inc.  Data Types: Aerial Photos and Archive Search, Field Inspections and Interviews  Data Collection Dates: February 1993 through October 1995	Historical information was used as reference.	None.
Final Verification Study	Originating Organization: HLA  Report Title: <i>Final Verification Report, Naval Construction Battalion Center, Gulfport, Mississippi</i>  Date: July 7, 1988	Originating Organization: HLA  Data Types: Evaluation of Previous Soil Sampling  Data Collection Dates: March 1987 through May 1987	Geophysical survey and analytical data will be used as reference.	Spacing for the electromagnetic survey was too wide; therefore, the geophysical survey will be used as reference for the upcoming survey. Also, the analytical data is not recent.

## **SAP Worksheet #14 – Summary of Project Tasks** **(UFP-QAPP Manual Section 2.8.1)**

### **14.1 SUMMARY OF PROJECT TASKS**

Sampling tasks include the following:

- Mobilization/demobilization
- Health and safety training
- Utility clearance
- Geophysical survey
- Passive soil gas screening
- Landfill gas survey
- Soil boring/subsurface soil sampling
- Groundwater sampling
- Surface water and sediment sampling
- Field decontamination procedure
- Investigation derived waste (IDW) management
- Documentation and records
- Data packages
- Data review tasks

#### **14.1.1 Mobilization and Demobilization**

Mobilization shall consist of the delivery of equipment, materials, and supplies to the site; the complete assembly in satisfactory working order of equipment at the site; and the satisfactory storage of materials and supplies at the site. Tetra Tech will coordinate with NCBC Gulfport to identify locations for the storage of equipment and supplies.

The fieldwork for the RI consists of four events; therefore, various selective mobilizations and demobilizations are anticipated. The results from the first three events will be used to shape the location, number, and type of samples collected in each subsequent event. For example, during Event 2 it is anticipated that 49 GORE-SORBER<sup>®</sup> Modules will be installed in a grid pattern over Site 2 (see Figure 6). The locations of the soil and groundwater samples in Event 3 will be based upon the results from the GORE-SORBER<sup>®</sup> Modules and the Event 1 geophysical survey.

A brief description of the field events is as follows:

- Event 1 – Geophysical Survey
- Event 2 – Passive Soil Gas Survey, Landfill Gas Survey, Ditch and Pond Investigation
- Event 3 – Soil and Groundwater Sampling
- Event 4 – Monitoring Well Installation and Additional Sampling as Needed

Demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Final demobilization includes the cleanup and removal of waste generated during the conduction of the investigation.

#### **14.1.2 Health and Safety Training**

Site-specific health and safety training per the site-specific HASP (Tetra Tech, 2009) will be provided to all Tetra Tech field crew and subcontractors as part of the site mobilization.

#### **14.1.3 Utility Clearance**

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate with Mississippi One-Call for utility locations. Mississippi One-Call will identify and mark-out utilities that may be present within the soil sampling locations.

#### **14.1.4 Event 1 – Geophysical Survey**

The geophysical survey will consist of an electromagnetic (EM) survey. It is anticipated that an EM-31 terrain conductivity meter and a magnetometer will be used. EM instruments measure the electrical conductivity and the relative metallic content of subsurface materials. Electrical conductivities are typically higher in areas containing buried waste. To perform the survey, a series of parallel profile lines will be established across the site at 10-foot intervals. The EM instrument will be moved along each of the lines, and measurements of terrain conductivity will be recorded at fixed distance intervals. The acquired data will be contoured and overlain onto an existing map of the site. The map will be used to identify terrain conductivity anomalies of the type commonly associated with buried waste. Location control will be provided using a global positioning system. Profile line spacing was chosen to provide sufficient resolution for delineation of areas of buried waste. Tetra Tech SOP GH-3.1, which describes EM survey procedures, is included in Appendix D. The information obtained during Event 1 will be communicated to the Project team and will be used to shape the activities planned for Event 2.

#### **14.1.5 Event 2 – Passive Soil Gas Screening**

Soil gas sampling involves the collection of vapors from soil pore spaces in the vadose zone that are collected and analyzed to determine the presence and concentration of materials capable of partitioning into the vapor phase under ambient conditions such as VOCs and lighter molecular weight SVOCs. Passive soil gas sampling involves the use of sampling units housing an adsorbent that are deployed in the subsurface for a specified period. The sampling units are retrieved and analyzed. During deployment, organic vapors migrating through the subsurface are passively collected onto the adsorbent. It is anticipated that GORE-SORBER® Modules will be used. Grid-based sample locations as presented on Figure 6 will be used.

#### **14.1.6 Event 2 – Landfill Gas Survey**

The landfill gas survey will collect and analyze gases from soil pore spaces for the presence of typical constituents generated by landfills. Temporary probe points will be installed at select locations. A landfill gas meter will be used to extract and analyze the soil gases. The landfill gas meter analyzes for methane (percent), oxygen, and carbon dioxide. Additionally, a multi-gas meter (PID) will be used to analyze for hydrogen sulfide, carbon monoxide, and VOCs. The sample locations presented on Figure 6 will be used. The information obtained during Events 1 and 2 will be communicated to the Project team and will be used to shape the activities planned for Event 3.

#### **14.1.7 Event 3 – Soil Boring/Subsurface Soil Sampling**

Biased sampling for soil will be based on the locations of geophysical anomalies and any passive soil gas detections of VOCs above MDEQ TRGs. Ten soil borings will be installed, and soil cores will be collected continuously from the ground surface to approximately 40 feet bls. The soil will be described by the site geologist and will be screened for evidence of contamination with a PID. Any qualitative signs of potential contamination (such as odor or staining) will be noted. Furthermore, depending on the preliminary results of the chemical data gathered during Event 3, additional subsurface sampling (not to exceed eight additional subsurface soil samples) will be collected in biased sample locations on Event 4. Soil sampling procedures are discussed in Tetra Tech SOP SA-1.3, soil logging procedures are discussed in Tetra Tech SOP GH-1.5, the use of the PID is discussed in Tetra Tech SOP ME-12. Field SOPs are included in Appendix D.

#### **14.1.8 Event 3 – Groundwater Sampling**

Ten groundwater samples will be taken during the soil boring activities from each of the soil boring locations mentioned above. Samples will be collected according to Tetra Tech SOPs SA-1.1 and GH-1.5,

and field screening will occur according to Tetra Tech SOP SF-1.3. Additionally, the protocol for temporary wells described in R.61-71, Mississippi Well Standards, April 26, 2002, will be followed.

#### **14.1.9 Event 3 – Surface Water and Sediment Sampling**

Sediment samples will be collected at each of the two outfalls in the western ditch as indicated in the aerial pictures and two sediments samples co-located with surface water samples will be collected from the deepest points in the manmade pond located on the eastern portion of the site as shown on Figure 6. Additionally, the ditch will be probed to determine if it is concrete lined. If the ditch is not lined with concrete, approximately five sediment samples will be taken from the bottom of the ditch in the centerline every 200 feet as shown on Figure 6. Samples will be collected according to Tetra Tech SOP SA-1-2. The information obtained during Events 1, 2, and 3 will be communicated to the Project Team and will be used to shape the activities planned for Event 4.

#### **14.1.10 Event 4 – Monitoring Well Installation and Additional Sampling as Needed**

Additionally, depending on the preliminary results of the chemical data gathered, additional groundwater samples (not to exceed 18) will be collected from newly installed monitoring wells. The location and depth of the newly installed wells will be based upon the findings from the first three investigation events. Samples will be collected according to Tetra Tech SOPs SA-1.1 and GH-1.5, and field screening will occur according to Tetra Tech SOP SF-1.3. Additionally, the protocol for temporary wells described in R.61-71, Mississippi Well Standards, April 26, 2002, will be followed.

Furthermore, based on the preliminary results of the chemical data gathered, additional surface water and sediment sampling may be conducted in based sample locations during Event 4. Samples will be collected according to Tetra Tech SOP SA-1-2.

#### **14.1.11 Field Decontamination Procedure**

Sample containers will be provided certified-clean from the analytical laboratories. Sampling equipment (e.g., non-disposable hand trowels, hand augers) will be decontaminated prior to and between sampling at each location. At each site, an abbreviated decontamination procedure consisting of a soapy water (laboratory-grade detergent) rinse followed by a deionized water rinse will be performed.

#### **14.1.12 Investigation Derived Waste Management**

It is anticipated waste materials will be generated during the field investigation. Wastes must be disposed in such a manner that does not contribute to further environmental contamination or pose a threat to public health or safety. Tetra Tech SOP SA-7.1 located in Appendix D provides information on the

handling of IDW. Drums for storage of IDW will be provided by NCBC Gulfport Public Works Division (PWD). Disposal of the IDW following receipt of the analytical data should be coordinated with the PWD.

#### **14.1.13 Documentation and Records**

Documentation of sample location coordinates, borings logs, chain-of-custody forms, samples logs, and shipping documents for samples will be recorded and filed. Preparation of electronic and hardcopies of the finalized Site 2 RI UFP SAP will be kept on site and in the Tetra Tech CTO 0150 project file.

#### **14.1.14 Data Packages**

Data packages will include the analytical data packages from the fixed-base laboratory, and generation of Tetra Tech data validation reports.

#### **14.1.15 Data Review Tasks**

The fixed-base laboratory will verify that all samples listed on the chain-of-custody are analyzed in accordance with methods specified on the chain-of-custody form, the laboratory scope of work, and in this SAP. Data verification and validation will be performed by Tetra Tech as described in Worksheets #35 and #36. A data validation report will be produced for each Sample Delivery Group (SDG).

The field data records and validated data will be reviewed by Tetra Tech personnel to determine the usability of the data (see Worksheet #37). The outcome of this assessment will be conveyed to the Project Team for agreement before the project report is finalized. Data limitations pertaining to Project Quality Objectives and PALs will be identified, and CAs will be taken as necessary.

**SAP Worksheet #15 – Reference Limits and Evaluation Table**  
 (UFP-QAPP Manual Section 2.8.1)

**Matrix: Soil**  
**Analytical: VOCs**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ACETONE	67-64-1	2.5	R5 ESL	0.02	0.01	0.005
<b>BENZENE</b>	<b>71-43-2</b>	<b>0.00023</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>BROMODICHLOROMETHANE</b>	<b>75-27-4</b>	<b>0.000033</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
BROMOFORM	75-25-2	0.0023	SSL	0.005	0.0025	0.00125
BROMOMETHANE	74-83-9	0.0022	SSL	0.01	0.005	0.0025
2-BUTANONE	78-93-3	1.5	SSL	0.01	0.005	0.0025
CARBON DISULFIDE	75-15-0	0.0941	R5 ESL	0.005	0.0025	0.00125
<b>CARBON TETRACHLORIDE</b>	<b>56-23-5</b>	<b>0.000079</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
CHLOROBENZENE	108-90-7	0.05	R4 ECO SOIL	0.005	0.0025	0.00125
CHLOROETHANE	75-00-3	6	SSL	0.01	0.005	0.0025
<b>CHLOROFORM</b>	<b>67-66-3</b>	<b>0.000055</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
CHLOROMETHANE	74-87-3	0.049	SSL	0.01	0.005	0.0025
<b>CHLORODIBROMOMETHANE</b>	<b>124-48-1</b>	<b>0.00004</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>1,2-DIBROMO-3-CHLOROPROPANE</b>	<b>96-12-8</b>	<b>0.00000015</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>1,2-DIBROMOETHANE</b>	<b>106-93-4</b>	<b>0.0000019</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
1,2-DICHLOROBENZENE	95-50-1	0.01	R4 ECO SOIL	0.005	0.0025	0.00125
1,3-DICHLOROBENZENE	541-73-1	0.01	R4 ECO SOIL	0.005	0.0025	0.00125
<b>1,4-DICHLOROBENZENE</b>	<b>106-46-7</b>	<b>0.00046</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>1,1-DICHLOROETHANE</b>	<b>75-34-3</b>	<b>0.0007</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>1,2-DICHLOROETHANE</b>	<b>107-06-2</b>	<b>0.000044</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
1,1-DICHLOROETHENE	75-35-4	0.0772	MS TIER 1 TRG	0.005	0.0025	0.00125
CIS-1,2-DICHLOROETHENE	156-59-2	0.11	SSL	0.005	0.0025	0.00125
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	150	SSL	0.005	0.0025	0.00125
TOTAL 1,2-DICHLOROETHENE	540-59-0	0.099	SSL	0.005	0.0025	0.00125
<b>CIS-1,3-DICHLOROPROPENE</b>	<b>10061-01-5</b>	<b>0.00016</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.398	R5 ESL	0.005	0.0025	0.00125
<b>ETHYLBENZENE</b>	<b>100-41-4</b>	<b>0.0019</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
2-HEXANONE	591-78-6	12.6	R5 ESL	0.01	0.005	0.0025
4-METHYL-2-PENTANONE	108-10-1	0.44	SSL	0.01	0.005	0.0025
<b>METHYLENE CHLORIDE</b>	<b>75-09-2</b>	<b>0.0012</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
STYRENE	100-42-5	0.1	R4 ECO SOIL	0.005	0.0025	0.00125

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
<b>1,1,2,2-TETRACHLOROETHANE</b>	<b>79-34-5</b>	<b>0.000028</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
1,1,1-TRICHLOROETHANE	71-55-6	3.3	SSL	0.005	0.0025	0.00125
<b>1,1,2-TRICHLOROETHANE</b>	<b>79-00-5</b>	<b>0.000082</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>TRICHLOROETHENE</b>	<b>79-01-6</b>	<b>0.00061</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>TETRACHLOROETHENE</b>	<b>127-18-4</b>	<b>0.000052</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
TOLUENE	108-88-3	0.05	R4 ECO SOIL	0.005	0.0025	0.00125
<b>VINYL CHLORIDE</b>	<b>75-01-4</b>	<b>0.0000056</b>	<b>SSL</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
TOTAL XYLENES	1330-20-7	0.05	R4 ECO SOIL	0.005	0.0025	0.00125
TRICHLOROFLUOROMETHANE	75-69-4	0.84	SSL	0.01	0.005	0.0025
DICHLORODIFLUOROMETHANE	75-71-8	0.61	SSL	0.01	0.005	0.0025

Notes:

CAS = Chemical Abstracts Service

mg/kg = Milligram per Kilogram

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Soil screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R-RSL = USEPA Regions 3, 6, and 9 Regional Screening Level (RSL), Direct Contact Residential (5/2010)

R4 ECO SOIL = USEPA Region 4 Ecological Soil Screening Values (11/2001)

R5 ESL = USEPA Region 5 Resource Conservation and Recovery Act (RCRA) Ecological Screening Level, Soil (8/2003)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

**Matrix: Soil**  
**Analytical: SVOCs and Low-Level PAHs\***

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
<b>1,2,4,5-TETRACHLOROBENZENE</b>	<b>95-94-3</b>	<b>0.01</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
1,1-BIPHENYL	92-52-4	23	SSL	0.333	0.167	0.083
2,4,5-TRICHLOROPHENOL	95-95-4	4	R4 ECO SOIL	0.333	0.167	0.083
<b>2,4,6-TRICHLOROPHENOL</b>	<b>88-06-2</b>	<b>0.016</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>2,4-DICHLOROPHENOL</b>	<b>120-83-2</b>	<b>0.003</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>2,4-DIMETHYLPHENOL</b>	<b>105-67-9</b>	<b>0.01</b>	<b>R5 ESL</b>	<b>1.33</b>	<b>0.667</b>	<b>0.333</b>
<b>2,4-DINITROPHENOL</b>	<b>51-28-5</b>	<b>0.068</b>	<b>SSL</b>	<b>3.3</b>	<b>1.67</b>	<b>0.83</b>
<b>2,4-DINITROTOLUENE</b>	<b>121-14-2</b>	<b>0.0002</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>2,6-DINITROTOLUENE</b>	<b>606-20-2</b>	<b>0.0328</b>	<b>R5 ESL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>2-CHLORONAPHTHALENE</b>	<b>91-58-7</b>	<b>0.0122</b>	<b>R5 ESL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
2-CHLOROPHENOL	95-57-8	0.2	SSL	0.333	0.167	0.083
2-METHYLNAPHTHALENE	91-57-6	0.9	SSL	0.333	0.167	0.083
2-METHYLPHENOL	95-48-7	0.5	USEPA ECO SSL	0.333	0.167	0.083
<b>2-NITROANILINE</b>	<b>88-74-4</b>	<b>0.033</b>	<b>SSL</b>	<b>1.33</b>	<b>0.667</b>	<b>0.333</b>
2-NITROPHENOL	88-75-5	7	R4 ECO SOIL	0.333	0.167	0.083
<b>2,2'-OXYBIS(1-CHLOROPROPANE)</b>	<b>108-60-1</b>	<b>0.00009</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>3,3'-DICHLOROBENZIDINE</b>	<b>91-94-1</b>	<b>0.0023</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
3-NITROANILINE	99-09-2	3.16	R5 ESL	1.33	0.667	0.333
<b>4,6-DINITRO-2-METHYLPHENOL</b>	<b>534-52-1</b>	<b>0.0051</b>	<b>SSL</b>	<b>3.3</b>	<b>01.67</b>	<b>0.83</b>
4-BROMOPHENYL PHENYL ETHER	101-55-3	---	---	0.333	0.167	0.083
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	---	---	0.333	0.167	0.083
4-CHLORO-3-METHYLPHENOL	59-50-7	7.95	R5 ESL	0.333	0.167	0.083
<b>4-CHLOROANILINE</b>	<b>106-47-8</b>	<b>0.00012</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
4-METHYLPHENOL	106-44-5	0.19	SSL	0.333	0.167	0.083
<b>4-NITROANILINE</b>	<b>100-01-6</b>	<b>0.001</b>	<b>SSL</b>	<b>1.3</b>	<b>0.667</b>	<b>0.333</b>
4-NITROPHENOL	100-02-7	7	R4 ECO SOIL	1.3	0.667	0.333
ACENAPHTHENE	83-32-9	27	SSL	0.01*	0.005*	0.002*
ACENAPHTHYLENE	208-96-8	27	SSL	0.01*	0.005*	0.002*
ACETOPHENONE	98-86-2	1.1	SSL	0.333	0.167	0.083
ANTHRACENE	120-12-7	29	USEPA ECO SSL	0.01*	0.005*	0.002*
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>0.00005</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>BENZALDEHYDE</b>	<b>100-52-7</b>	<b>0.97</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
BENZO(A)ANTHRACENE	56-55-3	0.014	SSL	0.01*	0.005*	0.002*

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
<b>BENZO(A)PYRENE</b>	<b>50-32-8</b>	<b>0.0046</b>	<b>SSL</b>	<b>0.01*</b>	<b>0.005*</b>	<b>0.002*</b>
BENZO(B)FLUORANTHENE	205-99-2	0.047	SSL	0.01*	0.005*	0.002*
BENZO(G,H,I)PERYLENE	191-24-2	1.1	USEPA ECO SSL	0.01*	0.005*	0.002*
BENZO(K)FLUORANTHENE	207-08-9	0.46	SSL	0.01*	0.005*	0.002*
<b>BIS(2-CHLOROETHOXY)METHANE</b>	<b>111-91-1</b>	<b>0.023</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>BIS(2-CHLOROETHYL)ETHER</b>	<b>111-44-4</b>	<b>0.0000027</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>BIS(2-ETHYLHEXYL)PHTHALATE</b>	<b>117-81-7</b>	<b>0.1</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>BUTYL BENZYL PHTHALATE</b>	<b>85-68-7</b>	<b>0.1</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
CAPROLACTAM	105-60-2	5.7	SSL	0.333	0.167	0.083
CARBAZOLE	86-74-8	31.9	MS TIER 1 TRG	0.333	0.167	0.083
CHRYSENE	218-01-9	1.1	USEPA ECO SSL	0.01*	0.005*	0.002*
DIBENZO(A,H)ANTHRACENE	53-70-3	0.015	R-RSL	0.01*	0.005*	0.002*
DIBENZOFURAN	132-64-9	313	MS TIER 1 TRG	0.333	0.167	0.083
DIETHYL PHTHALATE	84-66-2	13	SSL	0.333	0.167	0.083
DIMETHYL PHTHALATE	131-11-3	200	R4 ECO SOIL	0.333	0.167	0.083
DI-N-BUTYL PHTHALATE	84-74-2	11	SSL	0.333	0.167	0.083
<b>DI-N-OCTYL PHTHALATE</b>	<b>117-84-0</b>	<b>0.1</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
FLUORANTHENE	206-44-0	29	USEPA ECO SSL	0.01*	0.005*	0.002*
FLUORENE	86-73-7	29	USEPA ECO SSL	0.01*	0.005*	0.002*
<b>HEXACHLOROBENZENE</b>	<b>118-74-1</b>	<b>0.00029</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>HEXACHLOROBUTADIENE</b>	<b>87-68-3</b>	<b>0.0019</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
HEXACHLOROCYCLOPENTADIENE	77-47-4	0.8	SSL	0.333	0.167	0.083
<b>HEXACHLOROETHANE</b>	<b>67-72-1</b>	<b>0.0032</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
INDENO(1,2,3-CD)PYRENE	193-39-5	0.15	R-RSL	0.01*	0.005*	0.002*
<b>ISOPHORONE</b>	<b>78-59-1</b>	<b>0.022</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>NAPHTHALENE</b>	<b>91-20-3</b>	<b>0.00055</b>	<b>SSL</b>	<b>0.01*</b>	<b>0.005*</b>	<b>0.002*</b>
<b>NITROBENZENE</b>	<b>98-95-3</b>	<b>0.000071</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.000011</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>N-NITROSODIPHENYLAMINE</b>	<b>86-30-6</b>	<b>0.17</b>	<b>SSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>PENTACHLOROPHENOL</b>	<b>87-86-5</b>	<b>0.0039</b>	<b>SSL</b>	<b>1.33</b>	<b>0.667</b>	<b>0.333</b>
PHENANTHRENE	85-01-8	29	USEPA ECO SSL	0.01*	0.005*	0.002*
<b>PHENOL</b>	<b>108-95-2</b>	<b>0.05</b>	<b>R4 ECO SOIL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
PYRENE	129-00-0	1.1	USEPA ECO SSL	0.01*	0.005*	0.002*

Notes:

\* - 8270D Low Level Full Scan SOP will be utilized for PAHs.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Soil screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R-RSL = USEPA Regions 3, 6, and 9 RSL, Direct Contact Residential (5/2010)

R4 ECO SOIL = USEPA Region 4 Ecological Soil Screening Values (11/2001)

R5 ESL = USEPA Region 5 RCRA Ecological Screening Level, Soil (8/2003)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

USEPA ECO SSL = USEPA Eco-SSLs (2003-2007)

**Matrix: Soil**  
**Analytical: Pesticides**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL Reference <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ALDRIN	309-00-2	0.00084	SSL	0.0007	0.00035	0.00017
<b>ALPHA-BHC</b>	<b>319-84-6</b>	<b>0.000074</b>	<b>SSL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
ALPHA-CHLORDANE	5103-71-9	0.033	SSL	0.0007	0.00035	0.00017
CHLORDANE	57-74-9	0.033	SSL	0.0007	0.00035	0.00017
<b>BETA-BHC</b>	<b>319-85-7</b>	<b>0.00026</b>	<b>SSL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
4,4'-DDE	72-55-9	0.021	USEPA ECO SSL	0.0007	0.00035	0.00017
4,4'-DDD	72-54-8	0.021	USEPA ECO SSL	0.0007	0.00035	0.00017
4,4'-DDT	50-29-3	0.021	USEPA ECO SSL	0.0007	0.00035	0.00017
<b>DELTA-BHC</b>	<b>319-86-8</b>	<b>0.000074</b>	<b>SSL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
<b>DIELDRIN</b>	<b>60-57-1</b>	<b>0.00009</b>	<b>SSL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
ENDOSULFAN I	959-98-8	0.1	R4 ECO SOIL	0.0007	0.00035	0.00017
ENDOSULFAN II	33213-65-9	0.1	R4 ECO SOIL	0.0007	0.00035	0.00017
ENDOSULFAN SULFATE	1031-07-8	0.0358	R5 ESL	0.0007	0.00035	0.00017
ENDRIN	72-20-8	0.001	R4 ECO SOIL	0.0007	0.00035	0.00017
ENDRIN ALDEHYDE	7421-93-4	0.001	R4 ECO SOIL	0.0007	0.00035	0.00017
ENDRIN KETONE	53494-70-5	0.001	R4 ECO SOIL	0.0007	0.00035	0.00017
<b>GAMMA-BHC (LINDANE)</b>	<b>58-89-9</b>	<b>0.00005</b>	<b>R4 ECO SOIL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
GAMMA-CHLORDANE	5103-74-2	0.033	SSL	0.0007	0.00035	0.00017
HEPTACHLOR	76-44-8	0.0016	SSL	0.0007	0.00035	0.00017
<b>HEPTACHLOR EPOXIDE</b>	<b>1024-57-3</b>	<b>0.000079</b>	<b>SSL</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
METHOXYCHLOR	72-43-5	0.1	R4 ECO SOIL	0.0007	0.00035	0.00017
<b>TOXAPHENE</b>	<b>8001-35-2</b>	<b>0.012</b>	<b>SSL</b>	<b>0.033</b>	<b>0.022</b>	<b>0.011</b>

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Soil screening references:

R4 ECO SOIL = USEPA Region 4 Ecological Soil Screening Values (11/2001)

R5 ESL = USEPA Region 5 RCRA Ecological Screening Level, Soil (8/2003)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

USEPA ECO SSL = USEPA Eco-SSLs (2003-2007)

**Matrix: Soil**  
**Analytical: Herbicides**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
2,4,5-T	93-76-5	0.11	SSL	0.010	0.005	0.0025
2,4,5-TP (Silvex)	93-72-1	0.109	R5 ESL	0.010	0.005	0.0025
2,4-D	94-75-7	0.0272	R5 ESL	0.10	0.05	0.025

Notes:

<sup>1</sup> Soil screening references:

R5 ESL = USEPA Region 5 RCRA Ecological Screening Level, Soil (8/2003)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

**Matrix: Soil**  
**Analytical: PCBs**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
AROCLOR-1016	12674-11-2	0.052	R4 ECO SOIL	0.017	0.008	0.004
<b>AROCLOR-1221</b>	<b>11104-28-2</b>	<b>0.00014</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>
<b>AROCLOR-1232</b>	<b>11141-16-5</b>	<b>0.00014</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>
<b>AROCLOR-1242</b>	<b>53469-21-9</b>	<b>0.003</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>
<b>AROCLOR-1248</b>	<b>12672-29-6</b>	<b>0.003</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>
<b>AROCLOR-1254</b>	<b>11097-69-1</b>	<b>0.0051</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>
<b>AROCLOR-1260</b>	<b>11096-82-5</b>	<b>0.014</b>	<b>SSL</b>	<b>0.017</b>	<b>0.008</b>	<b>0.004</b>

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Soil screening references:

R4 ECO SOIL = USEPA Region 4 Ecological Soil Screening Values (11/2001)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

**Matrix: Soil**  
**Analytical: Inorganics (Metals and Cyanide)**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ALUMINUM	7429-90-5	50	R4 ECO SOIL	10	5	2.5
<b>ANTIMONY</b>	<b>7440-36-0</b>	<b>0.27</b>	<b>USEPA ECO SSL</b>	<b>0.75</b>	<b>0.4</b>	<b>0.25</b>
<b>ARSENIC</b>	<b>7440-38-2</b>	<b>0.0013</b>	<b>SSL</b>	<b>0.3</b>	<b>0.3</b>	<b>0.15</b>
BARIUM	7440-39-3	300	SSL	2	.5	.25
BERYLLIUM	7440-41-7	21	USEPA ECO SSL	0.25	0.1	0.05
CADMIUM	7440-43-9	0.36	USEPA ECO SSL	0.25	0.1	0.05
CALCIUM	7440-70-2	---	---	250	100	50
CHROMIUM	7440-47-3	26	USEPA ECO SSL	0.25	0.20	0.10
<b>COBALT</b>	<b>7440-48-4</b>	<b>0.49</b>	<b>SSL</b>	<b>0.63</b>	<b>0.50</b>	<b>0.25</b>
COPPER	7440-50-8	28	USEPA ECO SSL	0.5	0.4	0.25
IRON	7439-89-6	200	R4 ECO SOIL	5	3	1.5
LEAD	7439-92-1	11	USEPA ECO SSL	0.15	0.15	0.075
MAGNESIUM	7439-95-4	---	---	250	150	50
MANGANESE	7439-96-5	57	SSL	0.75	0.3	0.15
<b>MERCURY</b>	<b>7439-97-6</b>	<b>0.03</b>	<b>SSL</b>	<b>0.03</b>	<b>0.026</b>	<b>0.013</b>
NICKEL	7440-02-0	38	USEPA ECO SSL	0.5	0.3	0.25
POTASSIUM	7440-09-7	---	---	250	150	50
SELENIUM	7782-49-2	0.52	USEPA ECO SSL	0.3	0.25	0.15
SILVER	7440-22-4	1.6	SSL	0.25	0.1	0.05
SODIUM	7440-23-5	---	---	250	150	50
<b>THALLIUM</b>	<b>7440-28-0</b>	<b>0.17</b>	<b>SSL</b>	<b>0.4</b>	<b>0.2</b>	<b>0.15</b>
VANADIUM	7440-62-2	7.8	USEPA ECO SSL	0.63	0.5	0.25
ZINC	7440-66-6	46	USEPA ECO SSL	1	0.5	0.25
CYANIDE	57-12-5	0.9	R4 ECO SOIL	0.25	0.20	0.125

Notes:

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<sup>1</sup> Soil screening references:

R4 ECO SOIL = USEPA Region 4 Ecological Soil Screening Values (11/2001)

SSL = USEPA Regions 3, 6, and 9 Migration to Groundwater Soil Screening Level (5/2010)

USEPA ECO SSL = USEPA Eco-SSLs (2003-2007)

**Matrix: Soil**  
**Analytical: Dioxins/Furans**

ANALYTE	CAS NUMBER	PAL (pg/g)	PAL REFERENCE <sup>1</sup>	APPL		
				LOQ (pg/g)	LOD (pg/g)	DL (pg/g)
<b>1,2,3,4,6,7,8,9-OCDD</b>	<b>3268-87-9</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>25</b>	<b>5.08</b>	<b>2.54</b>
1,2,3,4,6,7,8,9-OCDF	39001-02-0	38.6	R5 ESL	25	5.62	2.81
<b>1,2,3,4,6,7,8-HPCDD</b>	<b>35822-46-9</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>12.5</b>	<b>2.82</b>	<b>1.41</b>
1,2,3,4,6,7,8-HPCDF	67562-39-4	38.6	R5 ESL	12.5	2.32	1.16
1,2,3,4,7,8,9-HPCDF	55673-89-7	38.6	R5 ESL	12.5	3.84	1.92
<b>1,2,3,4,7,8-HXCDD</b>	<b>39227-28-6</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>12.5</b>	<b>2.96</b>	<b>1.48</b>
1,2,3,4,7,8-HXCDF	70648-26-9	38.6	R5 ESL	12.5	2.08	1.04
<b>1,2,3,6,7,8-HXCDD</b>	<b>57653-85-7</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>12.5</b>	<b>1.96</b>	<b>0.98</b>
1,2,3,6,7,8-HXCDF	57117-44-9	38.6	R5 ESL	12.5	2.6	1.3
<b>1,2,3,7,8,9-HXCDD</b>	<b>19408-74-3</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>12.5</b>	<b>2.64</b>	<b>1.32</b>
1,2,3,7,8,9-HXCDF	72918-21-9	38.6	R5 ESL	12.5	7.24	3.62
<b>1,2,3,7,8-PECDD</b>	<b>40321-76-4</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>12.5</b>	<b>2.54</b>	<b>1.27</b>
1,2,3,7,8-PECDF	57117-41-6	38.6	R5 ESL	12.5	1.82	0.91
2,3,4,6,7,8-HXCDF	60851-34-5	38.6	R5 ESL	12.5	7.54	3.77
2,3,4,7,8-PECDF	57117-31-4	8.52	MS TIER 1 TRG	<b>12.5</b>	<b>4.92</b>	<b>2.46</b>
<b>2,3,7,8-TCDD</b>	<b>1746-01-6</b>	<b>0.199</b>	<b>R5 ESL</b>	<b>5</b>	<b>0.76</b>	<b>0.38</b>
2,3,7,8-TCDF	51207-31-9	38.6	R5 ESL	5	1.06	0.53
TOTAL HPCDD	37871-00-4	---	---	12.5	2.82	1.41
TOTAL HPCDF	38998-75-3	---	---	12.5	3.84	1.92
TOTAL HXCDD	34465-46-8	---	---	12.5	2.96	1.48
TOTAL HXCDF	55684-94-1	---	---	12.5	7.54	3.77
TOTAL PECDD	36088-22-9	---	---	12.5	2.54	1.27
TOTAL PECDF	30402-15-4	---	---	12.5	1.82	0.91
TOTAL TCDD	41903-57-5	---	---	5	0.76	0.38
TOTAL TCDF	55722-27-5	---	---	5	1.06	0.53

Notes:

pg/g = picogram per gram

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

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<sup>1</sup> Soil screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R5 ESL = USEPA Region 5 RCRA Ecological Screening Level, Soil (8/2003)

**Matrix: Sediment**  
**Analytical: VOCs**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
<b>ACETONE</b>	<b>67-64-1</b>	<b>0.0087</b>	<b>2CHRONIC</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
BENZENE	71-43-2	0.057	USEPA ECO TOX	0.005	0.0025	0.00125
BROMODICHLOROMETHANE	75-27-4	0.28	R-RSL	0.005	0.0025	0.00125
BROMOFORM	75-25-2	0.65	2CHRONIC	0.005	0.0025	0.00125
BROMOMETHANE	74-83-9	2.97	MS TIER 1 TRG	0.01	0.005	0.0025
2-BUTANONE	78-93-3	0.27	2CHRONIC	0.01	0.005	0.0025
<b>CARBON DISULFIDE</b>	<b>75-15-0</b>	<b>0.00085</b>	<b>2CHRONIC</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
CARBON TETRACHLORIDE	56-23-5	0.047	2CHRONIC	0.005	0.0025	0.00125
CHLOROBENZENE	108-90-7	0.00842	R3 FW SD	0.005	0.0025	0.00125
CHLOROETHANE	75-00-3	220	MS TIER 1 TRG	0.01	0.005	0.0025
CHLOROFORM	67-66-3	0.022	2CHRONIC	0.005	0.0025	0.00125
CHLOROMETHANE	74-87-3	49.1	MS TIER 1 TRG	0.01	0.005	0.0025
CHLORODIBROMOMETHANE	124-48-1	0.7	R-RSL	0.005	0.0025	0.00125
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.0056	R-RSL	0.005	0.0025	0.00125
1,2-DIBROMOETHANE	106-93-4	0.00751	MS TIER 1 TRG	0.005	0.0025	0.00125
1,2-DICHLOROBENZENE	95-50-1	0.0165	R3 FW SD	0.005	0.0025	0.00125
1,3-DICHLOROBENZENE	541-73-1	1.7	2CHRONIC	0.005	0.0025	0.00125
1,4-DICHLOROBENZENE	106-46-7	0.34	2CHRONIC	0.005	0.0025	0.00125
1,1-DICHLOROETHANE	75-34-3	0.027	2CHRONIC	0.005	0.0025	0.00125
1,2-DICHLOROETHANE	107-06-2	0.25	2CHRONIC	0.005	0.0025	0.00125
1,1-DICHLOROETHENE	75-35-4	0.031	R3 FW SD	0.005	0.0025	0.00125
CIS-1,2-DICHLOROETHENE	156-59-2	0.4	2CHRONIC	0.005	0.0025	0.00125
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	43,000	R-RSL	0.005	0.0025	0.00125
TOTAL 1,2-DICHLOROETHENE	540-59-0	0.4	2CHRONIC	0.005	0.0025	0.00125
<b>CIS-1,3-DICHLOROPROPENE</b>	<b>10061-01-5</b>	<b>0.000051</b>	<b>2CHRONIC</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
<b>TRANS-1,3-DICHLOROPROPENE</b>	<b>10061-02-6</b>	<b>0.000051</b>	<b>2CHRONIC</b>	<b>0.005</b>	<b>0.0025</b>	<b>0.00125</b>
ETHYLBENZENE	100-41-4	0.089	2CHRONIC	0.005	0.0025	0.00125
2-HEXANONE	591-78-6	0.022	2CHRONIC	0.01	0.005	0.0025
4-METHYL-2-PENTANONE	108-10-1	0.033	2CHRONIC	0.01	0.005	0.0025
METHYLENE CHLORIDE	75-09-2	0.37	2CHRONIC	0.005	0.0025	0.00125
STYRENE	100-42-5	0.559	R3 FW SD SSL	0.005	0.0025	0.00125
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.59	R-RSL	0.005	0.0025	0.00125
1,1,1-TRICHLOROETHANE	71-55-6	0.03	2CHRONIC	0.005	0.0025	0.00125
1,1,2-TRICHLOROETHANE	79-00-5	1.09	MS TIER 1 TRG	0.005	0.0025	0.00125

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TRICHLOROETHENE	79-01-6	0.0969	R3 FW SD	0.005	0.0025	0.00125
TETRACHLOROETHENE	127-18-4	0.41	2CHRONIC	0.005	0.0025	0.00125
TOLUENE	108-88-3	0.05	2CHRONIC	0.005	0.0025	0.00125
VINYL CHLORIDE	75-01-4	0.06	R-RSL	0.005	0.0025	0.00125
TOTAL XYLENES	1330-20-7	0.026	USEPA ECO TOX	0.005	0.0025	0.00125
TRICHLOROFLUOROMETHANE	75-69-4	800	R-RSL	0.01	0.005	0.0025
DICHLORODIFLUOROMETHANE	75-71-8	190	R-RSL	0.01	0.005	0.0025

Notes:

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Sediment screening references:

2CHRONIC = Secondary Chronic Criteria (Suter and Tsao, 1996)

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R-RSL = USEPA Regions 3, 6, and 9 RSL, Direct Contact Residential (5/2010)

R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

USEPA ECO TOX = USEPA's Ecological SSLs (2003-2007)

**Matrix: Sediment**  
**Analytical: SVOCs and Low-Level PAHs\***

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,2,4,5-TETRACHLOROBENZENE	95-94-3	1.09	R3 FW SD	0.333	0.167	0.083
1,1-BIPHENYL	92-52-4	1.1	2CHRONIC	0.333	0.167	0.083
2,4,5-TRICHLOROPHENOL	95-95-4	6,100	R-RSL	0.333	0.167	0.083
<b>2,4,6-TRICHLOROPHENOL</b>	<b>88-06-2</b>	<b>0.213</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
2,4-DICHLOROPHENOL	120-83-2	0.117	R3 FW SD	0.333	0.167	0.083
<b>2,4-DIMETHYLPHENOL</b>	<b>105-67-9</b>	<b>0.029</b>	<b>R3 FW SD</b>	<b>1.33</b>	<b>0.667</b>	<b>0.333</b>
2,4-DINITROPHENOL	51-28-5	120	R-RSL	3.3	1.67	0.83
<b>2,4-DINITROTOLUENE</b>	<b>121-14-2</b>	<b>0.0416</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
2,6-DINITROTOLUENE	606-20-2	61	R-RSL	0.333	0.167	0.083
2-CHLORONAPHTHALENE	91-58-7	6,260	MS TIER 1 TRG	0.333	0.167	0.083
<b>2-CHLOROPHENOL</b>	<b>95-57-8</b>	<b>0.0312</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
2-METHYLNAPHTHALENE	91-57-6	0.0202	R3 FW SD	0.333	0.167	0.083
<b>2-METHYLPHENOL</b>	<b>95-48-7</b>	<b>0.012</b>	<b>2CHRONIC</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
2-NITROANILINE	88-74-4	0.492	MS TIER 1 TRG	1.33	0.667	0.333
2-NITROPHENOL	88-75-5	---	---	0.333	0.167	0.083
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	3.5	R-RSL	0.333	0.167	0.083
<b>3,3'-DICHLOROBENZIDINE</b>	<b>91-94-1</b>	<b>0.127</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
3-NITROANILINE	99-09-2	---	---	1.33	0.667	0.333
4,6-DINITRO-2-METHYLPHENOL	534-52-1	6.1	R-RSL	3.3	01.67	0.83
4-BROMOPHENYL PHENYL ETHER	101-55-3	1.2	2CHRONIC	0.333	0.167	0.083
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	---	---	0.333	0.167	0.083
4-CHLORO-3-METHYLPHENOL	59-50-7	156,000	MS TIER 1 TRG	0.333	0.167	0.083
4-CHLOROANILINE	106-47-8	2.4	R-RSL	0.333	0.167	0.083
4-METHYLPHENOL	106-44-5	0.67	R3 FW SD	0.333	0.167	0.083
4-NITROANILINE	100-01-6	24	R-RSL	1.3	0.667	0.333
4-NITROPHENOL	100-02-7	626	MS TIER 1 TRG	1.3	0.667	0.333
<b>ACENAPHTHENE</b>	<b>83-32-9</b>	<b>0.0067</b>	<b>R3 FW SD</b>	<b>0.01</b>	<b>0.005</b>	<b>0.002</b>
<b>ACENAPHTHYLENE</b>	<b>208-96-8</b>	<b>0.00587</b>	<b>R4 SD EFFECT</b>	<b>0.01</b>	<b>0.005</b>	<b>0.002</b>
ACETOPHENONE	98-86-2	2,630	MS TIER 1 TRG	0.333	0.167	0.083
ANTHRACENE	120-12-7	0.0469	R4 SD EFFECT	0.01	0.005	0.002
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>0.00662</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
BENZALDEHYDE	100-52-7	7,800	R-RSL	0.333	0.167	0.083
BENZO(A)ANTHRACENE	56-55-3	0.0748	R4 SD EFFECT	0.01*	0.005*	0.002*
BENZO(A)PYRENE	50-32-8	0.015	R-RSL	0.01*	0.005*	0.002*

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
BENZO(B)FLUORANTHENE	205-99-2	0.15	R-RSL	0.01*	0.005*	0.002*
BENZO(G,H,I)PERYLENE	191-24-2	0.17	R3 FW SD	0.01*	0.005*	0.002*
BENZO(K)FLUORANTHENE	207-08-9	0.24	R3 FW SD	0.01*	0.005*	0.002*
BIS(2-CHLOROETHOXY)METHANE	111-91-1	180	R-RSL	0.333	0.167	0.083
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.19	R-RSL	0.333	0.167	0.083
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	0.18	R3 FW SD	0.333	0.167	0.083
BUTYL BENZYL PHTHALATE	85-68-7	10.9	R3 FW SD	0.333	0.167	0.083
CAPROLACTAM	105-60-2	31,000	R-RSL	0.333	0.167	0.083
CARBAZOLE	86-74-8	31.9	MS TIER 1 TRG	0.333	0.167	0.083
CHRYSENE	218-01-9	0.108	R4 SD EFFECT	0.01*	0.005*	0.002*
<b>DIBENZO(A,H)ANTHRACENE</b>	<b>53-70-3</b>	<b>0.00622</b>	<b>R4 SD EFFECT</b>	0.01*	0.005*	0.002*
DIBENZOFURAN	132-64-9	0.415	R3 FW SD	0.333	0.167	0.083
DIETHYL PHTHALATE	84-66-2	0.6	2CHRONIC	0.333	0.167	0.083
DIMETHYL PHTHALATE	131-11-3	782,000	MS TIER 1 TRG	0.333	0.167	0.083
DI-N-BUTYL PHTHALATE	84-74-2	6.47	R3 FW SD SSL	0.333	0.167	0.083
DI-N-OCTYL PHTHALATE	117-84-0	1,560	MS TIER 1 TRG	0.333	0.167	0.083
FLUORANTHENE	206-44-0	0.113	R4 SD EFFECT	0.01*	0.005*	0.002*
FLUORENE	86-73-7	0.0212	R4 SD EFFECT	0.01*	0.005*	0.002*
<b>HEXACHLOROENZENE</b>	<b>118-74-1</b>	<b>0.02</b>	<b>R3 FW SD</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
<b>HEXACHLOROBUTADIENE</b>	<b>87-68-3</b>	<b>0.0882</b>	<b>MS TIER 1 TRG</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
HEXACHLOROCYCLOPENTADIENE	77-47-4	0.951	MS TIER 1 TRG	0.333	0.167	0.083
HEXACHLOROETHANE	67-72-1	1	2CHRONIC	0.333	0.167	0.083
INDENO(1,2,3-CD)PYRENE	193-39-5	0.017	R3 FW SD	0.01*	0.005*	0.002**
ISOPHORONE	78-59-1	510	R-RSL	0.333	0.167	0.083
NAPHTHALENE	91-20-3	0.0346	R4 SD EFFECT	0.01*	0.005*	0.002
NITROBENZENE	98-95-3	4.4	R-RSL	0.333	0.167	0.083
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.069</b>	<b>R-RSL</b>	<b>0.333</b>	<b>0.167</b>	<b>0.083</b>
N-NITROSODIPHENYLAMINE	86-30-6	2.68	R3 FW SD	0.333	0.167	0.083
PENTACHLOROPHENOL	87-86-5	0.504	R3 FW SD	1.33	0.667	0.333
PHENANTHRENE	85-01-8	0.0867	R4 SD EFFECT	0.01*	0.005*	0.002*
PHENOL	108-95-2	0.42	R3 FW SD	0.333	0.167	0.083
PYRENE	129-00-0	0.153	R4 SD EFFECT	0.01*	0.005*	0.002*

Notes:

\* - 8270D Low Level Full Scan SOP will be utilized for PAHs.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Sediment screening references:

2CHRONIC = Secondary Chronic Criteria (Suter and Tsao, 1996)

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R-RSL = USEPA Regions 3, 6, and 9 RSL, Direct Contact Residential (5/2010)

R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

R4 SD EFFECT = USEPA Region 4 Ecological Sediment Screening Values (11/2001)

**Matrix: Sediment**  
**Analytical: Pesticides**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ALDRIN	309-00-2	0.002	R3 FW SD	0.0007	0.00035	0.00017
ALPHA-BHC	319-84-6	0.006	R3 FW SD	0.0007	0.00035	0.00017
ALPHA-CHLORDANE	5103-71-9	1.6	R-RSL	0.0007	0.00035	0.00017
<b>CHLORDANE</b>	<b>57-74-9</b>	<b>0.0005</b>	<b>R4 SD EFFECT</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
BETA-BHC	319-85-7	0.005	R3 FW SD	0.0007	0.00035	0.00017
4,4'-DDE	72-55-9	0.00207	R4 SD EFFECT	0.0007	0.00035	0.00017
4,4'-DDD	72-54-8	0.00122	R4 SD EFFECT	0.0007	0.00035	0.00017
4,4'-DDT	50-29-3	0.00119	R4 SD EFFECT	0.0007	0.00035	0.00017
DELTA-BHC	319-86-8	0.077	R-RSL	0.0007	0.00035	0.00017
<b>DIELDRIN</b>	<b>60-57-1</b>	<b>0.00002</b>	<b>R4 SD EFFECT</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
ENDOSULFAN I	959-98-8	0.0029	R3 FW SD	0.0007	0.00035	0.00017
ENDOSULFAN II	33213-65-9	0.0055	2CHRONIC	0.0007	0.00035	0.00017
ENDOSULFAN SULFATE	1031-07-8	0.0054	R3 FW SD	0.0007	0.00035	0.00017
<b>ENDRIN</b>	<b>72-20-8</b>	<b>0.00002</b>	<b>R4 SD EFFECT</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
ENDRIN ALDEHYDE	7421-93-4	18	R-RSL	0.0007	0.00035	0.00017
ENDRIN KETONE	53494-70-5	18	R-RSL	0.0007	0.00035	0.00017
<b>GAMMA-BHC (LINDANE)</b>	<b>58-89-9</b>	<b>0.00032</b>	<b>R4 SD EFFECT</b>	<b>0.0007</b>	<b>0.00035</b>	<b>0.00017</b>
GAMMA-CHLORDANE	5103-74-2	1.6	R-RSL	0.0007	0.00035	0.00017
HEPTACHLOR	76-44-8	0.068	R3 FW SD	0.0007	0.00035	0.00017
HEPTACHLOR EPOXIDE	1024-57-3	0.00247	R3 FW SD	0.0007	0.00035	0.00017
METHOXYCHLOR	72-43-5	0.0187	R3 FW SD	0.0007	0.00035	0.00017
<b>TOXAPHENE</b>	<b>8001-35-2</b>	<b>0.0001</b>	<b>R3 FW SD</b>	<b>0.033</b>	<b>0.022</b>	<b>0.011</b>

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQ and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Sediment screening references:

2CHRONIC = Secondary Chronic Criteria (Suter and Tsao, 1996)

R-RSL = USEPA Regions 3, 6, and 9 RSL, Direct Contact Residential (5/2010)

R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

R4 SD EFFECT = USEPA Region 4 Ecological Sediment Screening Values (11/2001)

**Matrix: Sediment**  
**Analytical: Herbicides**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
2,4,5-T	93-76-5	12.3	R3 FW SD	0.010	0.005	0.0025
2,4,5-TP (Silvex)	93-72-1	0.675	R3 FW SD	0.010	0.005	0.0025
2,4-D	94-75-7	690	R3 FW SD	0.10	0.05	0.025

Notes:

<sup>1</sup> Sediment screening references:  
 R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

**Matrix: Sediment**  
**Analytical: PCBs**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
AROCLOR-1016	12674-11-2	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1221	11104-28-2	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1232	11141-16-5	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1242	53469-21-9	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1248	12672-29-6	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1254	11097-69-1	0.0216	R4 SD EFFECT	0.017	0.008	0.004
AROCLOR-1260	11096-82-5	0.0216	R4 SD EFFECT	0.017	0.008	0.004

Notes:

<sup>1</sup> Sediment screening references:  
 R4 SD EFFECT = USEPA Region 4 Ecological Sediment Screening Values (11/2001)

**Matrix: Sediment**  
**Analytical: Inorganics (Metals and Cyanide)**

ANALYTE	CAS NUMBER	PAL (mg/kg)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
ALUMINUM	7429-90-5	77,000	R-RSL	10	5	2.5
ANTIMONY	7440-36-0	2	R4 SD EFFECT	0.75	0.4	0.25
ARSENIC	7440-38-2	0.39	R-RSL	0.3	0.3	0.15
BARIUM	7440-39-3	5,480	MS TIER 1 TRG	2	.5	.25
BERYLLIUM	7440-41-7	156	MS TIER 1 TRG	0.25	0.1	0.05
CADMIUM	7440-43-9	0.676	R4 SD EFFECT	0.25	0.1	0.05
CALCIUM	7440-70-2	---	---	250	100	50
CHROMIUM	7440-47-3	43.4	R3 FW SD	0.25	0.20	0.10
COBALT	7440-48-4	23	R-RSL	<b>0.63</b>	<b>0.50</b>	<b>0.25</b>
COPPER	7440-50-8	18.7	R4 SD EFFECT	0.5	0.4	0.25
IRON	7439-89-6	20,000	R3 FW SD	5	3	1.5
LEAD	7439-92-1	30.2	R4 SD EFFECT	0.15	0.15	0.075
MAGNESIUM	7439-95-4	---	---	250	150	50
MANGANESE	7439-96-5	460	R3 FW SD	0.75	0.3	0.15
MERCURY	7439-97-6	0.13	R4 SD EFFECT	0.03	0.026	0.013
NICKEL	7440-02-0	15.9	R4 SD EFFECT	0.5	0.3	0.25
POTASSIUM	7440-09-7	---	---	250	150	50
SELENIUM	7782-49-2	2	R3 FW SD	0.3	0.25	0.15
SILVER	7440-22-4	0.733	R4 SD EFFECT	0.25	0.1	0.05
SODIUM	7440-23-5	---	---	250	150	50
THALLIUM	7440-28-0	5.1	R-RSL	0.4	0.2	0.15
VANADIUM	7440-62-2	390	R-RSL	0.63	0.5	0.25
ZINC	7440-66-6	121	R3 FW SD	1	0.5	0.25
<b>CYANIDE</b>	<b>57-12-5</b>	<b>0.1</b>	<b>R3 FW SD</b>	<b>0.25</b>	<b>0.20</b>	<b>0.125</b>

Notes:

**Bolded and Shaded** compounds have LOQ and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Sediment screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R-RSL = USEPA Regions 3, 6, and 9 RSL, Direct Contact Residential (5/2010)

R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

R4 SD EFFECT = USEPA Region 4 Ecological Sediment Screening Values (11/2001)

**Matrix: Sediment**  
**Analytical: Dioxins/Furans**

ANALYTE	CAS NUMBER	PAL (pg/g)	PAL REFERENCE <sup>1</sup>	APPL		
				LOQ (pg/g)	LOD (pg/g)	DL (pg/g)
1,2,3,4,6,7,8,9-OCDD	3268-87-9	4,260	MS TIER 1 TRG	25	5.08	2.54
1,2,3,4,6,7,8,9-OCDF	39001-02-0	4,260	MS TIER 1 TRG	25	5.62	2.81
1,2,3,4,6,7,8-HPCDD	35822-46-9	426	MS TIER 1 TRG	12.5	2.82	1.41
1,2,3,4,6,7,8-HPCDF	67562-39-4	426	MS TIER 1 TRG	12.5	2.32	1.16
1,2,3,4,7,8,9-HPCDF	55673-89-7	426	MS TIER 1 TRG	12.5	3.84	1.92
1,2,3,4,7,8-HXCDD	39227-28-6	42.6	MS TIER 1 TRG	12.5	2.96	1.48
1,2,3,4,7,8-HXCDF	70648-26-9	42.6	MS TIER 1 TRG	12.5	2.08	1.04
1,2,3,6,7,8-HXCDD	57653-85-7	103	MS TIER 1 TRG	12.5	1.96	0.98
1,2,3,6,7,8-HXCDF	57117-44-9	42.6	MS TIER 1 TRG	12.5	2.6	1.3
1,2,3,7,8,9-HXCDD	19408-74-3	103	MS TIER 1 TRG	12.5	2.64	1.32
1,2,3,7,8,9-HXCDF	72918-21-9	42.6	MS TIER 1 TRG	12.5	7.24	3.62
<b>1,2,3,7,8-PECDD</b>	<b>40321-76-4</b>	<b>8.52</b>	<b>MS TIER 1 TRG</b>	<b>12.5</b>	<b>2.54</b>	<b>1.27</b>
1,2,3,7,8-PECDF	57117-41-6	85.2	MS TIER 1 TRG	12.5	1.82	0.91
2,3,4,6,7,8-HXCDF	60851-34-5	42.6	MS TIER 1 TRG	12.5	7.54	3.77
<b>2,3,4,7,8-PECDF</b>	<b>57117-31-4</b>	<b>8.52</b>	<b>MS TIER 1 TRG</b>	<b>12.5</b>	<b>4.92</b>	<b>2.46</b>
<b>2,3,7,8-TCDD</b>	<b>1746-01-6</b>	<b>0.85</b>	<b>R3 FW SD</b>	<b>5</b>	<b>0.76</b>	<b>0.38</b>
2,3,7,8-TCDF	51207-31-9	42.6	MS TIER 1 TRG	5	1.06	0.53
TOTAL HPCDD	37871-00-4	---	---	12.5	2.82	1.41
TOTAL HPCDF	38998-75-3	---	---	12.5	3.84	1.92
TOTAL HXCDD	34465-46-8	---	---	12.5	2.96	1.48
TOTAL HXCDF	55684-94-1	---	---	12.5	7.54	3.77
TOTAL PECDD	36088-22-9	---	---	12.5	2.54	1.27
TOTAL PECDF	30402-15-4	---	---	12.5	1.82	0.91
TOTAL TCDD	41903-57-5	---	---	5	0.76	0.38
TOTAL TCDF	55722-27-5	---	---	5	1.06	0.53

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

<sup>1</sup> Sediment screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Unrestricted Soil (2/2002)

R3 FW SD = USEPA Region 3 Ecological Freshwater Sediment Screening Benchmarks (8/2006)

**Matrix: Groundwater**  
**Analytical: VOCs**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
ACETONE	67-64-1	608	MS TIER 1 TRG	10	5	2.5
<b>BENZENE</b>	<b>71-43-2</b>	<b>0.41</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>BROMODICHLOROMETHANE</b>	<b>75-27-4</b>	<b>0.12</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
BROMOFORM	75-25-2	8.48	MS TIER 1 TRG	2	1	.5
BROMOMETHANE	74-83-9	8.52	MS TIER 1 TRG	1	0.5	0.25
2-BUTANONE	78-93-3	1,910	MS TIER 1 TRG	10	5	2.5
CARBON DISULFIDE	75-15-0	1,000	T-RSL	1	0.5	0.25
<b>CARBON TETRACHLORIDE</b>	<b>56-23-5</b>	<b>0.2</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
CHLOROBENZENE	108-90-7	91	T-RSL	1	0.5	0.25
CHLOROETHANE	75-00-3	3.64	MS TIER 1 TRG	1	0.5	0.25
<b>CHLOROFORM</b>	<b>67-66-3</b>	<b>0.155</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
CHLOROMETHANE	74-87-3	1.43	MS TIER 1 TRG	1	0.5	0.25
<b>CHLORODIBROMOMETHANE</b>	<b>124-48-1</b>	<b>0.126</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>1,2-DIBROMO-3-CHLOROPROPANE</b>	<b>96-12-8</b>	<b>0.00032</b>	<b>T-RSL</b>	<b>2</b>	<b>1</b>	<b>0.5</b>
<b>1,2-DIBROMOETHANE</b>	<b>106-93-4</b>	<b>0.0065</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,2-DICHLOROBENZENE	95-50-1	370	T-RSL	1	0.5	0.25
1,3-DICHLOROBENZENE	541-73-1	5.48	MS TIER 1 TRG	1	0.5	0.25
<b>1,4-DICHLOROBENZENE</b>	<b>106-46-7</b>	<b>0.43</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1-DICHLOROETHANE	75-34-3	2.4	T-RSL	1	0.5	0.25
<b>1,2-DICHLOROETHANE</b>	<b>107-06-2</b>	<b>0.15</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1-DICHLOROETHENE	75-35-4	7	MS TIER 1 TRG	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	MS TIER 1 TRG	1	0.5	0.25
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	59,000	T-RSL	1	0.5	0.25
TOTAL 1,2-DICHLOROETHENE	540-59-0	330	T-RSL	2	1	0.5
<b>CIS-1,3-DICHLOROPROPENE</b>	<b>10061-01-5</b>	<b>0.43</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TRANS-1,3-DICHLOROPROPENE	10061-02-6	---	---	1	0.5	0.25
ETHYLBENZENE	100-41-4	1.5	T-RSL	1	0.5	0.25
2-HEXANONE	591-78-6	1,460	MS TIER 1 TRG	5	2.5	1.25
4-METHYL-2-PENTANONE	108-10-1	139	MS TIER 1 TRG	10	5	2.5
METHYLENE CHLORIDE	75-09-2	4.8	T-RSL	4	2	1
STYRENE	100-42-5	100	MS TIER 1 TRG	1	0.5	0.25
<b>1,1,2,2-TETRACHLOROETHANE</b>	<b>79-34-5</b>	<b>0.0527</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1,1-TRICHLOROETHANE	71-55-6	200	MS TIER 1 TRG	1	0.5	0.25

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>1,1,2-TRICHLOROETHANE</b>	<b>79-00-5</b>	<b>0.24</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TRICHLOROETHENE	79-01-6	1.7	T-RSL	1	0.5	0.25
<b>TETRACHLOROETHENE</b>	<b>127-18-4</b>	<b>0.11</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TOLUENE	108-88-3	1,000	MS TIER 1 TRG	1	0.5	0.25
<b>VINYL CHLORIDE</b>	<b>75-01-4</b>	<b>0.016</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TOTAL XYLENES	1330-20-7	200	T-RSL	3	2	1
TRICHLOROFLUOROMETHANE	75-69-4	1,290	MS TIER 1 TRG	1	0.5	0.25
DICHLORODIFLUOROMETHANE	75-71-8	348	MS TIER 1 TRG	1	0.5	0.25

Notes:

µg/L = microgram per liter

**Bolded and Shaded** compounds have LOQ and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Groundwater screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Groundwater**  
**Analytical: SVOCs and Low-Level PAHs\***

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,2,4,5-TETRACHLOROBENZENE	95-94-3	11	MS TIER 1 TRG	5	2.5	1.25
1,1-BIPHENYL	92-52-4	304	MS TIER 1 TRG	5	2.5	1.25
2,4,5-TRICHLOROPHENOL	95-95-4	3,650	MS TIER 1 TRG	5	2.5	1.25
2,4,6-TRICHLOROPHENOL	88-06-2	6.09	MS TIER 1 TRG	5	2.5	1.25
2,4-DICHLOROPHENOL	120-83-2	110	MS TIER 1 TRG	5	2.5	1.25
2,4-DIMETHYLPHENOL	105-67-9	730	MS TIER 1 TRG	20	10	5
2,4-DINITROPHENOL	51-28-5	73	MS TIER 1 TRG	50	25	10
<b>2,4-DINITROTOLUENE</b>	<b>121-14-2</b>	<b>0.22</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
2,6-DINITROTOLUENE	606-20-2	36.5	MS TIER 1 TRG	5	2.5	1.25
2-CHLORONAPHTHALENE	91-58-7	487	MS TIER 1 TRG	5	2.5	1.25
2-CHLOROPHENOL	95-57-8	30.4	MS TIER 1 TRG	5	2.5	1.25
2-METHYLNAPHTHALENE	91-57-6	122	MS TIER 1 TRG	5	2.5	1.25
2-METHYLPHENOL	95-48-7	1,800	T-RSL	5	2.5	1.25
<b>2-NITROANILINE</b>	<b>88-74-4</b>	<b>0.417</b>	<b>MS TIER 1 TRG</b>	<b>20</b>	<b>10</b>	<b>5</b>
<b>2-NITROPHENOL</b>	<b>88-75-5</b>	<b>0.416</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>2,2'-OXYBIS(1-CHLOROPROPANE)</b>	<b>108-60-1</b>	<b>0.26</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>3,3'-DICHLOROBENZIDINE</b>	<b>91-94-1</b>	<b>0.149</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
3-NITROANILINE	99-09-2	---	---	20	10	5
<b>4,6-DINITRO-2-METHYLPHENOL</b>	<b>534-52-1</b>	<b>3.65</b>	<b>MS TIER 1 TRG</b>	<b>20</b>	<b>10</b>	<b>5</b>
4-BROMOPHENYL PHENYL ETHER	101-55-3	---	---	5	2.5	1.25
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	---	---	5	2.5	1.25
4-CHLORO-3-METHYLPHENOL	59-50-7	73,000	MS TIER 1 TRG	5	2.5	1.25
<b>4-CHLOROANILINE</b>	<b>106-47-8</b>	<b>0.34</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
4-METHYLPHENOL	106-44-5	180	T-RSL	5	2.5	1.25
<b>4-NITROANILINE</b>	<b>100-01-6</b>	<b>3.4</b>	<b>T-RSL</b>	<b>20</b>	<b>10</b>	<b>5</b>
4-NITROPHENOL	100-02-7	292	MS TIER 1 TRG	20	10	5
ACENAPHTHENE	83-32-9	365	MS TIER 1 TRG	0.2*	0.1*	0.05*
ACENAPHTHYLENE	208-96-8	2,190	MS TIER 1 TRG	0.2*	0.1*	0.05*
<b>ACETOPHENONE</b>	<b>98-86-2</b>	<b>0.0416</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
ANTHRACENE	120-12-7	43.4	MS TIER 1 TRG	0.2	0.1	0.05
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>0.29</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
BENZALDEHYDE	100-52-7	3,650	MS TIER 1 TRG	5	2.5	1.25
<b>BENZO(A)ANTHRACENE</b>	<b>56-55-3</b>	<b>0.029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>BENZO(A)PYRENE</b>	<b>50-32-8</b>	<b>0.0029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>BENZO(B)FLUORANTHENE</b>	<b>205-99-2</b>	<b>0.029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
BENZO(G,H,I)PERYLENE	191-24-2	1,100	MS TIER 1 TRG	0.2*	0.1*	0.05*
<b>BENZO(K)FLUORANTHENE</b>	<b>207-08-9</b>	<b>0.29</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
BIS(2-CHLOROETHOXY)METHANE	111-91-1	110	T-RSL	5	2.5	1.25
<b>BIS(2-CHLOROETHYL)ETHER</b>	<b>111-44-4</b>	<b>0.0092</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>BIS(2-ETHYLHEXYL)PHTHALATE</b>	<b>117-81-7</b>	<b>4.8</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
BUTYL BENZYL PHTHALATE	85-68-7	35	T-RSL	5	2.5	1.25
CAPROLACTAM	105-60-2	18,000	T-RSL	5	2.5	1.25
<b>CARBAZOLE</b>	<b>86-74-8</b>	<b>3.35</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
CHRYSENE	218-01-9	2.9	T-RSL	0.2*	0.1*	0.05*
<b>DIBENZO(A,H)ANTHRACENE</b>	<b>53-70-3</b>	<b>0.0029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
DIBENZOFURAN	132-64-9	24.3	MS TIER 1 TRG	5	2.5	1.25
DIETHYL PHTHALATE	84-66-2	29,000	T-RSL	5	2.5	1.25
DIMETHYL PHTHALATE	131-11-3	365,000	MS TIER 1 TRG	5	2.5	1.25
DI-N-BUTYL PHTHALATE	84-74-2	3650	MS TIER 1 TRG	5	2.5	1.25
DI-N-OCTYL PHTHALATE	117-84-0	20	MS TIER 1 TRG	5	2.5	1.25
FLUORANTHENE	206-44-0	1,460	MS TIER 1 TRG	0.2*	0.1*	0.05*
FLUORENE	86-73-7	243	MS TIER 1 TRG	0.2*	0.1*	0.05*
<b>HEXACHLOROBENZENE</b>	<b>118-74-1</b>	<b>0.042</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>HEXACHLOROBUTADIENE</b>	<b>87-68-3</b>	<b>0.859</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
HEXACHLOROCYCLOPENTADIENE	77-47-4	50	MS TIER 1 TRG	5	2.5	1.25
HEXACHLOROETHANE	67-72-1	4.78	MS TIER 1 TRG	5	2.5	1.25
<b>INDENO(1,2,3-CD)PYRENE</b>	<b>193-39-5</b>	<b>0.029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
ISOPHORONE	78-59-1	70.5	MS TIER 1 TRG	5	2.5	1.25
<b>NAPHTHALENE</b>	<b>91-20-3</b>	<b>0.14</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>NITROBENZENE</b>	<b>98-95-3</b>	<b>0.12</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.00957</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
N-NITROSODIPHENYLAMINE	86-30-6	13.7	MS TIER 1 TRG	5	2.5	1.25
<b>PENTACHLOROPHENOL</b>	<b>87-86-5</b>	<b>0.56</b>	<b>T-RSL</b>	<b>20</b>	<b>10</b>	<b>5</b>
PHENANTHRENE	85-01-8	1,100	MS TIER 1 TRG	0.2*	0.1*	0.05*
PHENOL	108-95-2	11,000	T-RSL	5	2.5	1.25
PYRENE	129-00-0	183	MS TIER 1 TRG	5	2.5	1.25

Notes:

\* - 8270D Low Level Full Scan SOP will be utilized for PAHs.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Groundwater screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Groundwater**  
**Analytical: Pesticides**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>ALDRIN</b>	<b>309-00-2</b>	<b>0.00394</b>	<b>MS TIER 1 TRG</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>ALPHA-BHC</b>	<b>319-84-6</b>	<b>0.0106</b>	<b>MS TIER 1 TRG</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
ALPHA-CHLORDANE	5103-71-9	0.19	T-RSL	0.02	0.01	0.005
CHLORDANE	57-74-9	0.19	T-RSL	0.05	0.025	0.0125
BETA-BHC	319-85-7	0.037	T-RSL	0.02	0.01	0.005
4,4'-DDE	72-55-9	0.197	MS TIER 1 TRG	0.02	0.01	0.005
4,4'-DDD	72-54-8	0.279	MS TIER 1 TRG	0.02	0.01	0.005
4,4'-DDT	50-29-3	0.197	MS TIER 1 TRG	0.02	0.01	0.005
<b>DELTA-BHC</b>	<b>319-86-8</b>	<b>0.011</b>	<b>T-RSL</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>DIELDRIN</b>	<b>60-57-1</b>	<b>0.00419</b>	<b>MS TIER 1 TRG</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
ENDOSULFAN I	959-98-8	220	T-RSL	0.02	0.01	0.005
ENDOSULFAN II	33213-65-9	220	T-RSL	0.02	0.01	0.005
ENDOSULFAN SULFATE	1031-07-8	220	T-RSL	0.02	0.01	0.005
ENDRIN	72-20-8	2	MS TIER 1 TRG	0.02	0.01	0.005
ENDRIN ALDEHYDE	7421-93-4	11	T-RSL	0.02	0.01	0.005
ENDRIN KETONE	53494-70-5	11	T-RSL	0.02	0.01	0.005
GAMMA-BHC (LINDANE)	58-89-9	0.061	T-RSL	0.02	0.01	0.005
GAMMA-CHLORDANE	5103-74-2	0.19	T-RSL	0.02	0.01	0.005
HEPTACHLOR	76-44-8	0.015	T-RSL	0.02	0.01	0.005
<b>HEPTACHLOR EPOXIDE</b>	<b>1024-57-3</b>	<b>0.0074</b>	<b>T-RSL</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
METHOXYCHLOR	72-43-5	40	MS TIER 1 TRG	0.02	0.01	0.005
<b>TOXAPHENE</b>	<b>8001-35-2</b>	<b>0.061</b>	<b>T-RSL</b>	<b>1.0</b>	<b>0.667</b>	<b>0.333</b>

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Groundwater screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Groundwater**  
**Analytical: Herbicides**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
2,4,5-T	93-76-5	365	MS TIER 1 TRG	0.10	0.05	0.025
2,4,5-TP (Silvex)	93-72-1	50	MS TIER 1 TRG	0.10	0.05	0.025
2,4-D	94-75-7	70	MS TIER 1 TRG	1.0	0.5	0.25

Notes:

<sup>1</sup> Groundwater screening references:  
 MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

**Matrix: Groundwater**  
**Analytical: PCBs**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
AROCLOR-1016	12674-11-2	0.96	T-RSL	0.5	0.25	0.125
<b>AROCLOR-1221</b>	<b>11104-28-2</b>	<b>0.0068</b>	T-RSL	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1232</b>	<b>11141-16-5</b>	<b>0.0068</b>	T-RSL	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1242</b>	<b>53469-21-9</b>	<b>0.0335</b>	MS TIER 1 TRG	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1248</b>	<b>12672-29-6</b>	<b>0.0335</b>	MS TIER 1 TRG	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1254</b>	<b>11097-69-1</b>	<b>0.0335</b>	MS TIER 1 TRG	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1260</b>	<b>11096-82-5</b>	<b>0.0335</b>	MS TIER 1 TRG	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>

Notes:

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Groundwater screening references:

MCL = USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (5/2009)

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Groundwater**  
**Analytical: Inorganics (Metals and Cyanide)**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
ALUMINUM	7429-90-5	36,500	MS TIER 1 TRG	200	100	50
ANTIMONY (1)	7440-36-0	6	MS TIER 1 TRG	4	2	1
<b>ARSENIC (1)</b>	<b>7440-38-2</b>	<b>0.045</b>	<b>T-RSL</b>	<b>1.5</b>	<b>1.5</b>	<b>0.75</b>
BARIUM	7440-39-3	2,000	MS TIER 1 TRG	40	20	10
BERYLLIUM	7440-41-7	4	MS TIER 1 TRG	4	2	1
CADMIUM	7440-43-9	5	MS TIER 1 TRG	5	2	1
CALCIUM	7440-70-2	---	---	5,000	2,000	1,000
CHROMIUM	7440-47-3	100	MCL	10	4	2
COBALT	7440-48-4	11	T-RSL	11	10	5
COPPER	7440-50-8	1,300	MS TIER 1 TRG	10	8	4
IRON	7439-89-6	11,000	MS TIER 1 TRG	100	60	30
LEAD	7439-92-1	15	MS TIER 1 TRG	3	3	1.5
MAGNESIUM	7439-95-4	---	---	5,000	3,000	1,000
MANGANESE	7439-96-5	730	MS TIER 1 TRG	15	10	5
MERCURY	7439-97-6	0.57	T-RSL	0.2	0.16	0.08
NICKEL	7440-02-0	730	MS TIER 1 TRG	10	6	3
POTASSIUM	7440-09-7	---	---	5,000	3,000	1,000
SELENIUM	7782-49-2	50	MS TIER 1 TRG	6	5	3
SILVER	7440-22-4	180	T-RSL	10	2	1
SODIUM	7440-23-5	---	---	5,000	3,000	1,000
THALLIUM (1)	7440-28-0	2	MS TIER 1 TRG	2	1	0.75
VANADIUM	7440-62-2	180	T-RSL	12.5	10	5
ZINC	7440-66-6	11,000	MS TIER 1 TRG	20	10	5
CYANIDE	57-12-5	200	MS TIER 1 TRG	0.01	0.0075	0.005

Notes:

(1) Empirical will concentrate 4X per USEPA 200.7 to obtain lower detection limits for these analytes.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Groundwater screening references:

MCL = USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (5/2009)

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Groundwater**  
**Analytical: Dioxins/Furans**

ANALYTE	CAS NUMBER	PAL (pg/L)	PAL REFERENCE <sup>1</sup>	APPL		
				LOQ (pg/L)	LOD (pg/L)	DL (pg/L)
1,2,3,4,6,7,8,9-OCDD	3268-87-9	446	MS TIER 1 TRG	250	41.78	20.89
1,2,3,4,6,7,8,9-OCDF	39001-02-0	446	MS TIER 1 TRG	250	64.04	32.02
<b>1,2,3,4,6,7,8-HPCDD</b>	<b>35822-46-9</b>	<b>44.6</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>31.16</b>	<b>15.58</b>
<b>1,2,3,4,6,7,8-HPCDF</b>	<b>67562-39-4</b>	<b>44.6</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>23.82</b>	<b>11.91</b>
<b>1,2,3,4,7,8,9-HPCDF</b>	<b>55673-89-7</b>	<b>44.6</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>53.82</b>	<b>26.91</b>
<b>1,2,3,4,7,8-HXCDD</b>	<b>39227-28-6</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>29.32</b>	<b>14.66</b>
<b>1,2,3,4,7,8-HXCDF</b>	<b>70648-26-9</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>13.22</b>	<b>6.61</b>
<b>1,2,3,6,7,8-HXCDD</b>	<b>57653-85-7</b>	<b>10.8</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>37.64</b>	<b>18.82</b>
<b>1,2,3,6,7,8-HXCDF</b>	<b>57117-44-9</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>15.08</b>	<b>7.54</b>
<b>1,2,3,7,8,9-HXCDD</b>	<b>19408-74-3</b>	<b>10.8</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>57.22</b>	<b>28.61</b>
<b>1,2,3,7,8,9-HXCDF</b>	<b>72918-21-9</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>27.06</b>	<b>13.53</b>
<b>1,2,3,7,8-PECDD</b>	<b>40321-76-4</b>	<b>0.89</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>30.24</b>	<b>15.12</b>
<b>1,2,3,7,8-PECDF</b>	<b>57117-41-6</b>	<b>8.9</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>15.78</b>	<b>7.89</b>
<b>2,3,4,6,7,8-HXCDF</b>	<b>60851-34-5</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>91.22</b>	<b>45.6</b>
<b>2,3,4,7,8-PECDF</b>	<b>57117-31-4</b>	<b>0.89</b>	<b>MS TIER 1 TRG</b>	<b>125</b>	<b>38.08</b>	<b>19.04</b>
<b>2,3,7,8-TCDD</b>	<b>1746-01-6</b>	<b>30</b>	<b>MS TIER 1 TRG</b>	<b>50</b>	<b>14.9</b>	<b>7.45</b>
<b>2,3,7,8-TCDF</b>	<b>51207-31-9</b>	<b>4.5</b>	<b>MS TIER 1 TRG</b>	<b>50</b>	<b>10.06</b>	<b>5.03</b>
TOTAL HPCDD	37871-00-4	---	---	125	31.16	15.58
TOTAL HPCDF	38998-75-3	---	---	125	53.82	26.91
TOTAL HXCDD	34465-46-8	---	---	125	57.22	28.61
TOTAL HXCDF	55684-94-1	---	---	125	91.22	45.6
TOTAL PECDD	36088-22-9	---	---	125	30.24	15.12
TOTAL PECDF	30402-15-4	---	---	125	38.08	19.04
TOTAL TCDD	41903-57-5	---	---	50	14.9	7.45
TOTAL TCDF	55722-27-5	---	---	50	10.06	5.03

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup> Groundwater screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

**Matrix: Surface Water**  
**Analytical: VOCs**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
ACETONE	67-64-1	608	MS TIER 1 TRG	10	5	2.5
<b>BENZENE</b>	<b>71-43-2</b>	<b>0.41</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>BROMODICHLOROMETHANE</b>	<b>75-27-4</b>	<b>0.12</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
BROMOFORM	75-25-2	8.48	MS TIER 1 TRG	2	1	0.5
BROMOMETHANE	74-83-9	8.52	MS TIER 1 TRG	1	0.5	0.25
2-BUTANONE	78-93-3	1,910	MS TIER 1 TRG	10	5	2.5
<b>CARBON DISULFIDE</b>	<b>75-15-0</b>	<b>0.92</b>	<b>R3 FW SW</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>CARBON TETRACHLORIDE</b>	<b>56-23-5</b>	<b>0.2</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
CHLOROENZENE	108-90-7	91	T-RSL	1	0.5	0.25
CHLOROETHANE	75-00-3	3.64	MS TIER 1 TRG	1	0.5	0.25
<b>CHLOROFORM</b>	<b>67-66-3</b>	<b>0.155</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
CHLOROMETHANE	74-87-3	1.43	MS TIER 1 TRG	1	0.5	0.25
<b>CHLORODIBROMOMETHANE</b>	<b>124-48-1</b>	<b>0.126</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>1,2-DIBROMO-3-CHLOROPROPANE</b>	<b>96-12-8</b>	<b>0.00032</b>	<b>T-RSL</b>	<b>2</b>	<b>1</b>	<b>0.5</b>
<b>1,2-DIBROMOETHANE</b>	<b>106-93-4</b>	<b>0.0065</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,2-DICHLOROENZENE	95-50-1	15.8	R4 FW SW	1	0.5	0.25
1,3-DICHLOROENZENE	541-73-1	5.48	MS TIER 1 TRG	1	0.5	0.25
<b>1,4-DICHLOROENZENE</b>	<b>106-46-7</b>	<b>0.43</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1-DICHLOROETHANE	75-34-3	2.4	T-RSL	1	0.5	0.25
<b>1,2-DICHLOROETHANE</b>	<b>107-06-2</b>	<b>0.15</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1-DICHLOROETHENE	75-35-4	7	MS TIER 1 TRG	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	MS TIER 1 TRG	1	0.5	0.25
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	59,000	T-RSL	1	0.5	0.25
TOTAL 1,2-DICHLOROETHENE	540-59-0	330	T-RSL	2	1	0.5
<b>CIS-1,3-DICHLOROPROPENE</b>	<b>10061-01-5</b>	<b>0.43</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TRANS-1,3-DICHLOROPROPENE	10061-02-6	24.4	R4 FW SW	1	0.5	0.25
ETHYLBENZENE	100-41-4	1.5	T-RSL	1	0.5	0.25
2-HEXANONE	591-78-6	99	R3 FW SW	5	2.5	1.25
4-METHYL-2-PENTANONE	108-10-1	139	MS TIER 1 TRG	10	5	2.5
METHYLENE CHLORIDE	75-09-2	4.8	T-RSL	4	2	1
STYRENE	100-42-5	72	R3 FW SW	1	0.5	0.25
<b>1,1,2,2-TETRACHLOROETHANE</b>	<b>79-34-5</b>	<b>0.0527</b>	<b>MS TIER 1 TRG</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1,1-TRICHLOROETHANE	71-55-6	200	MS TIER 1 TRG	1	0.5	0.25
<b>1,1,2-TRICHLOROETHANE</b>	<b>79-00-5</b>	<b>0.24</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TRICHLOROETHENE	79-01-6	1.7	T-RSL	1	0.5	0.25

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>TETRACHLOROETHENE</b>	<b>127-18-4</b>	<b>0.11</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TOLUENE	108-88-3	175	R4 FW SW	1	0.5	0.25
<b>VINYL CHLORIDE</b>	<b>75-01-4</b>	<b>0.016</b>	<b>T-RSL</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TOTAL XYLENES	1330-20-7	13	R3 FW SW	3	2	1
TRICHLOROFLUOROMETHANE	75-69-4	1,290	MS TIER 1 TRG	1	0.5	0.25
DICHLORODIFLUOROMETHANE	75-71-8	348	MS TIER 1 TRG	1	0.5	0.25

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Surface Water screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

R3 FW SW = USEPA Region 3 Ecological Freshwater Screening Benchmarks (7/2006)

R4 FW SW = USEPA Region 4 Ecological Freshwater Surface Water Screening Values (11/2001)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Surface Water**  
**Analytical: SVOCs and Low-Level PAHs\***

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,2,4,5-TETRACHLORO BENZENE	95-94-3	11	MS TIER 1 TRG	5	2.5	1.25
1,1-BIPHENYL	92-52-4	14	R3 FW SW	5	2.5	1.25
2,4,5-TRICHLOROPHENOL	95-95-4	3,650	MS TIER 1 TRG	5	2.5	1.25
<b>2,4,6-TRICHLOROPHENOL</b>	<b>88-06-2</b>	<b>3.2</b>	<b>R4 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
2,4-DICHLOROPHENOL	120-83-2	36.5	R4 FW SW	5	2.5	1.25
2,4-DIMETHYLPHENOL	105-67-9	21.2	R4 FW SW	20	10	5
<b>2,4-DINITROPHENOL</b>	<b>51-28-5</b>	<b>6.2</b>	<b>R4 FW SW</b>	<b>50</b>	<b>25</b>	<b>10</b>
<b>2,4-DINITROTOLUENE</b>	<b>121-14-2</b>	<b>0.22</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
2,6-DINITROTOLUENE	606-20-2	36.5	MS TIER 1 TRG	5	2.5	1.25
2-CHLORONAPHTHALENE	91-58-7	487	MS TIER 1 TRG	5	2.5	1.25
2-CHLOROPHENOL	95-57-8	30.4	MS TIER 1 TRG	5	2.5	1.25
<b>2-METHYLNAPHTHALENE</b>	<b>91-57-6</b>	<b>4.7</b>	<b>R3 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
2-METHYLPHENOL	95-48-7	13	R3 FW SW	5	2.5	1.25
<b>2-NITROANILINE</b>	<b>88-74-4</b>	<b>0.417</b>	<b>MS TIER 1 TRG</b>	<b>20</b>	<b>10</b>	<b>5</b>
<b>2-NITROPHENOL</b>	<b>88-75-5</b>	<b>0.416</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>2,2'-OXYBIS(1-CHLOROPROPANE)</b>	<b>108-60-1</b>	<b>0.26</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>3,3'-DICHLOROBENZIDINE</b>	<b>91-94-1</b>	<b>0.149</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
3-NITROANILINE	99-09-2	---	---	20	10	5
<b>4,6-DINITRO-2-METHYLPHENOL</b>	<b>534-52-1</b>	<b>2.3</b>	<b>R4 FW SW</b>	<b>20</b>	<b>10</b>	<b>5</b>
4-BROMOPHENYL PHENYL ETHER	101-55-3	12.2	R4 FW SW	5	2.5	1.25
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	---	---	5	2.5	1.25
<b>4-CHLORO-3-METHYLPHENOL</b>	<b>59-50-7</b>	<b>0.3</b>	<b>R4 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>4-CHLOROANILINE</b>	<b>106-47-8</b>	<b>0.34</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
4-METHYLPHENOL	106-44-5	180	T-RSL	5	2.5	1.25
<b>4-NITROANILINE</b>	<b>100-01-6</b>	<b>3.4</b>	<b>T-RSL</b>	<b>20</b>	<b>10</b>	<b>5</b>
4-NITROPHENOL	100-02-7	82.8	R4 FW SW	20	10	5
ACENAPHTHENE	83-32-9	17	R4 FW SW	0.2*	0.1*	0.05*
ACENAPHTHYLENE	208-96-8	2,190	MS TIER 1 TRG	0.2*	0.1*	0.05*
<b>ACETOPHENONE</b>	<b>98-86-2</b>	<b>0.0416</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>ANTHRACENE</b>	<b>120-12-7</b>	<b>0.012</b>	<b>R3 FW SW</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>0.29</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
BENZALDEHYDE	100-52-7	3,650	MS TIER 1 TRG	5	2.5	1.25
<b>BENZO(A)ANTHRACENE</b>	<b>56-55-3</b>	<b>0.018</b>	<b>R3 FW SW</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>BENZO(A)PYRENE</b>	<b>50-32-8</b>	<b>0.0029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>BENZO(B)FLUORANTHENE</b>	<b>205-99-2</b>	<b>0.029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
BENZO(G,H,I)PERYLENE	191-24-2	1,100	MS TIER 1 TRG	0.2*	0.1*	0.05*
BENZO(K)FLUORANTHENE	207-08-9	0.29	T-RSL	0.2*	0.1*	0.05*
BIS(2-CHLOROETHOXY)METHANE	111-91-1	110	T-RSL	5	2.5	1.25
<b>BIS(2-CHLOROETHYL)ETHER</b>	<b>111-44-4</b>	<b>0.0092</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>BIS(2-ETHYLHEXYL)PHTHALATE</b>	<b>117-81-7</b>	<b>0.3</b>	<b>R4 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
BUTYL BENZYL PHTHALATE	85-68-7	22	R4 FW SW	5	2.5	1.25
CAPROLACTAM	105-60-2	18,000	T-RSL	5	2.5	1.25
<b>CARBAZOLE</b>	<b>86-74-8</b>	<b>3.35</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
CHRYSENE	218-01-9	2.9	T-RSL	0.2*	0.1*	0.05*
<b>DIBENZO(A,H)ANTHRACENE</b>	<b>53-70-3</b>	<b>0.0029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>DIBENZOFURAN</b>	<b>132-64-9</b>	<b>3.7</b>	<b>R3 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
DIETHYL PHTHALATE	84-66-2	521	R4 FW SW	5	2.5	1.25
DIMETHYL PHTHALATE	131-11-3	330	R4 FW SW	5	2.5	1.25
DI-N-BUTYL PHTHALATE	84-74-2	9.4	R4 FW SW	5	2.5	1.25
DI-N-OCTYL PHTHALATE	117-84-0	20	MS TIER 1 TRG	5	2.5	1.25
FLUORANTHENE	206-44-0	39.8	R4 FW SW	0.2*	0.1*	0.05*
FLUORENE	86-73-7	3	R3 FW SW	0.2*	0.1*	0.05*
<b>HEXACHLOROBENZENE</b>	<b>118-74-1</b>	<b>0.0003</b>	<b>R3 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>HEXACHLOROBUTADIENE</b>	<b>87-68-3</b>	<b>0.859</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>HEXACHLOROCYCLOPENTADIENE</b>	<b>77-47-4</b>	<b>0.07</b>	<b>R4 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>HEXACHLOROETHANE</b>	<b>67-72-1</b>	<b>4.78</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>INDENO(1,2,3-CD)PYRENE</b>	<b>193-39-5</b>	<b>0.029</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
ISOPHORONE	78-59-1	70.5	MS TIER 1 TRG	5	2.5	1.25
<b>NAPHTHALENE</b>	<b>91-20-3</b>	<b>0.14</b>	<b>T-RSL</b>	<b>0.2*</b>	<b>0.1*</b>	<b>0.05*</b>
<b>NITROBENZENE</b>	<b>98-95-3</b>	<b>0.12</b>	<b>T-RSL</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.00957</b>	<b>MS TIER 1 TRG</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>
N-NITROSODIPHENYLAMINE	86-30-6	13.7	MS TIER 1 TRG	5	2.5	1.25
<b>PENTACHLOROPHENOL</b>	<b>87-86-5</b>	<b>0.56</b>	<b>T-RSL</b>	<b>20</b>	<b>10</b>	<b>5</b>
PHENANTHRENE	85-01-8	0.4	R3 FW SW	0.2*	0.1*	0.05*
PHENOL	108-95-2	256	R4 FW SW	5	2.5	1.25
<b>PYRENE</b>	<b>129-00-0</b>	<b>0.025</b>	<b>R3 FW SW</b>	<b>5</b>	<b>2.5</b>	<b>1.25</b>

Notes:

\* - 8270D Low Level Full Scan SOP will be utilized for PAHs.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Surface water screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

R3 FW SW = USEPA Region 3 Ecological Freshwater Screening Benchmarks (7/2006)

R4 FW SW = USEPA Region 4 Ecological Freshwater Surface Water Screening Values (11/2001)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Surface Water**  
**Analytical: Pesticides**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>ALDRIN</b>	<b>309-00-2</b>	<b>0.00394</b>	<b>MS TIER 1 TRG</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>ALPHA-BHC</b>	<b>319-84-6</b>	<b>0.0106</b>	<b>MS TIER 1 TRG</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>ALPHA-CHLORDANE</b>	<b>5103-71-9</b>	<b>0.0043</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>CHLORDANE</b>	<b>57-74-9</b>	<b>0.0043</b>	<b>R4 FW SW</b>	<b>0.05</b>	<b>0.025</b>	<b>0.0125</b>
BETA-BHC	319-85-7	0.037	T-RSL	0.02	0.01	0.005
4,4'-DDE	72-55-9	0.197	MS TIER 1 TRG	0.02	0.01	0.005
<b>4,4'-DDD</b>	<b>72-54-8</b>	<b>0.0064</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>4,4'-DDT</b>	<b>50-29-3</b>	<b>0.001</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>DELTA-BHC</b>	<b>319-86-8</b>	<b>0.011</b>	<b>T-RSL</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>DIELDRIN</b>	<b>60-57-1</b>	<b>0.0019</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
ENDOSULFAN I	959-98-8	0.056	R4 FW SW	0.02	0.01	0.005
ENDOSULFAN II	33213-65-9	0.056	R4 FW SW	0.02	0.01	0.005
ENDOSULFAN SULFATE	1031-07-8	0.05	R4 FW SW	0.02	0.01	0.005
<b>ENDRIN</b>	<b>72-20-8</b>	<b>0.0023</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>ENDRIN ALDEHYDE</b>	<b>7421-93-4</b>	<b>0.0023</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>ENDRIN KETONE</b>	<b>53494-70-5</b>	<b>0.0023</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
GAMMA-BHC (LINDANE)	58-89-9	0.061	T-RSL	0.02	0.01	0.005
<b>GAMMA-CHLORDANE</b>	<b>5103-74-2</b>	<b>0.0043</b>	<b>R3 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>HEPTACHLOR</b>	<b>76-44-8</b>	<b>0.0038</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
<b>HEPTACHLOR EPOXIDE</b>	<b>1024-57-3</b>	<b>0.0038</b>	<b>R4 FW SW</b>	<b>0.02</b>	<b>0.01</b>	<b>0.005</b>
METHOXYCHLOR	72-43-5	0.03	R4 FW SW	0.02	0.01	0.005
<b>TOXAPHENE</b>	<b>8001-35-2</b>	<b>0.0002</b>	<b>R4 FW SW</b>	<b>1.0</b>	<b>0.667</b>	<b>0.333</b>

Notes:

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Surface water screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

R3 FW SW = USEPA Region 3 Ecological Freshwater Screening Benchmarks (7/2006)

R4 FW SW = USEPA Region 4 Ecological Freshwater Surface Water Screening Values (11/2001)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Surface Water**  
**Analytical: Herbicides**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
2,4,5-T	93-76-5	365	MS TIER 1 TRG	0.10	0.05	0.025
2,4,5-TP (Silvex)	93-72-1	30	R3 FW SW	0.10	0.05	0.025
2,4-D	94-75-7	70	MS TIER 1 TRG	1.0	0.5	0.25

Notes:

<sup>1</sup>Surface water screening references:  
 MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)  
 R3 FW SW = USEPA Region 3 Ecological Freshwater Screening Benchmarks (7/2006)

**Matrix: Surface Water**  
**Analytical: PCBs**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>AROCLOR-1016</b>	<b>12674-11-2</b>	<b>0.014</b>	R4 FW SW	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1221</b>	<b>11104-28-2</b>	<b>0.0068</b>	T-RSL	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1232</b>	<b>11141-16-5</b>	<b>0.0068</b>	T-RSL	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1242</b>	<b>53469-21-9</b>	<b>0.014</b>	R4 FW SW	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1248</b>	<b>12672-29-6</b>	<b>0.014</b>	R4 FW SW	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1254</b>	<b>11097-69-1</b>	<b>0.014</b>	R4 FW SW	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>
<b>AROCLOR-1260</b>	<b>11096-82-5</b>	<b>0.014</b>	R4 FW SW	<b>0.5</b>	<b>0.25</b>	<b>0.125</b>

Notes:

**Bolded and Shaded** compounds have LOQ and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report. <sup>1</sup>Surface water screening references:

R4 FW SW – USEPA Region 4 Ecological Freshwater Surface Water Screening Values (11/2001)  
 T-RSL – USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

**Matrix: Surface Water**  
**Analytical: Inorganics (Metals and Cyanide)**

ANALYTE	CAS NUMBER	PAL (µg/L)	PAL REFERENCE <sup>1</sup>	EMPIRICAL		
				LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
<b>ALUMINUM</b>	<b>7429-90-5</b>	<b>87</b>	<b>R4 FW SW</b>	<b>200</b>	<b>100</b>	<b>50</b>
ANTIMONY (1)	7440-36-0	6	MS TIER 1 TRG	4	2	1
<b>ARSENIC (1)</b>	<b>7440-38-2</b>	<b>0.045</b>	<b>T-RSL</b>	<b>1.5</b>	<b>1.5</b>	<b>0.75</b>
<b>BARIUM</b>	<b>7440-39-3</b>	<b>4</b>	<b>R3 FW SW</b>	<b>40</b>	<b>20</b>	<b>10</b>
<b>BERYLLIUM</b>	<b>7440-41-7</b>	<b>0.53</b>	<b>R4 FW SW</b>	<b>4</b>	<b>2</b>	<b>1</b>
<b>CADMIUM</b>	<b>7440-43-9</b>	<b>0.66</b>	<b>R4 FW SW</b>	<b>5</b>	<b>2</b>	<b>1</b>
CALCIUM	7440-70-2	116,000	R3 FW SW	5,000	2,000	1,000
CHROMIUM	7440-47-3	11	R4 FW SW	10	4	2
COBALT	7440-48-4	11	T-RSL	11	10	5
<b>COPPER</b>	<b>7440-50-8</b>	<b>6.54</b>	<b>R4 FW SW</b>	<b>10</b>	<b>8</b>	<b>4</b>
IRON	7439-89-6	1,000	R4 FW SW	100	60	30
<b>LEAD</b>	<b>7439-92-1</b>	<b>1.32</b>	<b>R4 FW SW</b>	<b>3</b>	<b>3</b>	<b>1.5</b>
MAGNESIUM	7439-95-4	82,000	R3 FW SW	5,000	3,000	1,000
MANGANESE	7439-96-5	120	R3 FW SW	15	10	5
<b>MERCURY</b>	<b>7439-97-6</b>	<b>0.012</b>	<b>R4 FW SW</b>	<b>0.2</b>	<b>0.16</b>	<b>0.08</b>
NICKEL	7440-02-0	87.71	R4 FW SW	10	6	3
POTASSIUM	7440-09-7	53,000	R3 FW SW	5,000	3,000	1,000
<b>SELENIUM</b>	<b>7782-49-2</b>	<b>5</b>	<b>R4 FW SW</b>	<b>6</b>	<b>5</b>	<b>3</b>
<b>SILVER</b>	<b>7440-22-4</b>	<b>0.012</b>	<b>R4 FW SW</b>	<b>10</b>	<b>2</b>	<b>1</b>
SODIUM	7440-23-5	680,000	R3 FW SW	5,000	3,000	1,000
THALLIUM (1)	7440-28-0	2	MS TIER 1 TRG	2	1	0.75
VANADIUM	7440-62-2	20	R3 FW SW	12.5	10	5
ZINC	7440-66-6	58.91	R4 FW SW	20	10	5
CYANIDE	57-12-5	5.2	R4 FW SW	0.01	0.0075	0.005

Notes:

(1) Empirical will concentrate 4X per USEPA 200.7 to obtain lower detection limits for these analytes.

**Bolded** compounds indicate PAL values that are less than the laboratory LOQ. However, the LOD is sufficiently low to meet the PAL and for the intended data use.

**Bolded and Shaded** compounds have LOQs and LODs that do not meet the PAL. The approach for risk assessment and decision making is described in Worksheet #11, Sections 11.2 and 11.4. Any uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the RI Report.

<sup>1</sup>Surface water screening references:

MS TIER 1 TRG = MDEQ Tier 1 TRGs, Groundwater (2/2002)

T-RSL = USEPA Regions 3, 6, and 9 RSL, Tapwater (5/2010)

R3 FW SW = USEPA Region 3 Ecological Freshwater Screening Benchmarks (7/2006)

R4 FW SW = USEPA Region 4 Ecological Freshwater Surface Water Screening Values (11/2001)

**SAP Worksheet #16 -- Project Schedule / Timeline Table**  
 (UFP-QAPP Manual Section 2.8.2)

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE ACTUAL SUBMITTAL
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION	
Prepare Rough Draft SAP Work Plan and Appendices	Tetra Tech	07/01/09	12/18/09	
<b>Submit Rough Draft SAP Work Plan and Appendices</b>	Tetra Tech	---	12/22/09	
Navy Review	Navy	12/18/09	01/15/10	
Prepare Draft SAP Work Plan and Appendices	Tetra Tech	08/15/10	08/15/10	
<b>Submit Draft SAP Work Plan and Appendices</b>	Tetra Tech	---	08/15/10	
Regulator Review	MDEQ	08/16/10/10	09/01/10	
Receive Comments/Comment Resolution	Tetra Tech	09/01/10	09/03/10	
Prepare Final SAP Work Plan and Appendices	Tetra Tech	09/03/10	09/07/10	
<b>Submit Final SAP Work Plan &amp; Appendices</b>	Tetra Tech	---	09/10/10	
<b>Mobilization and Field Investigation</b>	Tetra Tech	09/13/10	02/28/11	
<b>Complete Field Investigation and Demobilization</b>	Tetra Tech	---	02/28/11	
<b>Laboratory Analysis</b>	Empirical and APPL	09/13/10	03/28/11	
<b>Data Validation</b>	Tetra Tech	11/30/10	05/18/11	
Database Entry	Tetra Tech	11/30/10	05/30/11	
Prepare Rough Draft Report	Tetra Tech	03/06/11	06/15/11	
<b>Submit Rough Draft Report</b>	Tetra Tech	---	06/15/11	
Navy Review	Navy	06/18/11	07/02/11	
Prepare Draft Report	Tetra Tech	07/03/11	07/13/11	
<b>Submit Draft Report</b>	Tetra Tech	---	07/13/11	
Regulator Review	MDEQ	07/16/11	09/08/11	
Receive Comments/Comment Resolution	Tetra Tech	09/08/11	09/22/11	
Prepare Final Report	Tetra Tech	09/08/11	09/29/11	
<b>Submit Final Report</b>	Tetra Tech	---	09/29/11	

**Bold** activities are deliverables.

## **SAP Worksheet #17 – Sampling Design and Rationale** **(UFP-QAPP Manual Section 3.1.1)**

### **17.1 INTRODUCTION**

This section describes sampling locations, methods, and rationales for the sampling activities to be conducted in support of the field investigations at Site 2 located at NCBC Gulfport. The referenced field SOPs and field forms are presented in Appendix D. The general rationale for the decisions identified in Worksheets #10 and #11 is presented in Sections 17.2 through 17.5. The methodology for sample collection and field screening of samples is presented in Worksheet #14. The analytical program recommended for each proposed sample is presented in Worksheet #18. The field QC samples required are specified in Worksheet #20. To the extent possible, the referenced sampling locations are depicted on Figures 6 and 6A.

The SAP presents a flexible and iterative approach to sampling. This approach builds upon the results from earlier investigations (see Worksheet 10) and seeks to address the data gaps resulting from those earlier investigations. The fieldwork for the RI consists of four events; i.e.; (1) Geophysical Survey, (2) Passive Soil Gas Survey, Landfill Gas Survey, Ditch and Pond Investigation, (3) Soil and Groundwater Sampling, and (4) Monitoring Well Installation and Additional Sampling as Needed (see Worksheet 14). Each event provides information that will be used in the next event to refine the location, number, and the type of sample collection points. For example, during Event 2, 49 GORE-SORBER<sup>®</sup> Modules will be installed in a grid pattern over Site 2 (see Figure 6). The locations of the soil and groundwater samples in Event 3 will be based upon the results from the GORE-SORBER<sup>®</sup> Modules and the Event 1 geophysical survey. Therefore, it is not possible at this time to show the anticipated soil and groundwater locations. However, the locations, numbers, and type of samples are intended to delineate fully the nature and extent of impacts for Site 2.

The criteria detailed below and discussed in Sections 17.2 through 17.5 should be met for a site to follow the presumptive remedy approach.

- Delineation of the Landfill Area: The presumptive remedy for a landfill is containment; therefore, the landfill area should be fully delineated to apply properly the suggested remedy.
- Delineation of Hotspots: One of the criteria for a presumptive remedy approach is that risks are low-level except for hotspots. During the field events, a passive soil gas survey will be used to screen for hotspots. Further analytical data from a fixed-base laboratory will be used to confirm the passive gas survey.

- **Characterization of Waste:** Multi-media sampling (subsurface soil, sediment, surface water, and groundwater) and visual observations in the field will complement and confirm historical information regarding the waste disposed at Site 2.
  - Waste types are generally household, commercial, non-hazardous sludge, and industrial solid wastes.
  - Lesser quantities of hazardous wastes are present as compared to municipal-type wastes, if any.
  - No hazard military-specific wastes (such as unexploded ordnance, radioactive waste, or biological/chemical warfare agents) are anticipated.
- **Evaluation of Migration Pathways:** Sediment and collocated surface water and groundwater samples will be collected to provide for information for hotspot delineation, and delineation of the landfill area.

## **17.2 DELINEATION OF LANDFILL AREA**

The boundaries of the waste disposal area(s) will be identified during the geophysical survey. Additionally, if waste materials are encountered during surface and subsurface soil sampling, the type and distribution of the waste material will be documented in the soil boring logs.

## **17.3 DETECTION OF HOT SPOTS**

The passive soil gas survey will utilize a PID for VOC screening at sample locations to identify potential hot spots. Further confirmation of potential hot spots will be performed by collecting groundwater and soil samples and sending the samples to a fixed-base laboratory to be analyzed for VOCs and SVOCs.

## **17.4 CHARACTERIZATION OF WASTE**

Approximately 10 soil and 10 groundwater samples will be collected from soil borings to determine the extent of contamination in environmental media at Site 2. Soil samples from borings will be collected continuously up to 40 feet bls and screened for VOCs with a PID. Soil samples selected (per the decision rule presented in Worksheet #11) from one depth interval will be submitted to Empirical (and to APPL for the soil and groundwater dioxins/furans) for analysis. The groundwater sample intervals will be based on the PID field screening and the lithologies observed during soil boring advancement. The proposed sampling locations will be distributed based on the geophysical survey and passive soil vapor analysis as explained in Worksheet #11. However, additional sampling locations may be collected based on information from the previous sampling phases. The additional groundwater locations (eight sample locations) will be taken from eight permanent wells (which will be installed according to the soil gas) and random soil and groundwater samples.

## 17.5 EVALUATION OF MIGRATION PATHWAYS

Four sediment samples (0 to 6 inches) and four surface water samples (collocated with the sediment samples) will be collected from the pond on the eastern side of Site 2 as shown on Figure 6. Additionally, five sediment samples may be taken from the centerline of the ditch located west of the site at 200-foot intervals. These samples will be collected to evaluate the potential migration of contaminants from the waste disposal area to the receiving water body. The lithology of the sediments observed during installation of sediment borings will be described in the field sampling logs. Water quality parameters will be measured and logged in the field for surface water samples. Sediment samples and surface water samples will be submitted to Empirical (and to APPL for dioxins/furans) for analysis. Based on the preliminary results, additional sediment and/or surface water samples (not to exceed 5 additional samples per media) could be collected.

Additional groundwater, sediment, and surface water samples may be collected from identified hot spots and from locations beyond the investigation perimeter for Site 2 to determine if contaminants released from the waste disposal area pose unacceptable human health risks or may be migrating beyond site boundaries. The monitoring well and additional sampling locations will be “biased or judgmental” sampling locations and will be selected by the lead hydrogeologist for Site 2 based on environmental data collected during the screening investigation and hydrogeological data currently available for Site 2. The monitoring well locations will be biased toward the location(s) demonstrating the maximum screening results.

As indicated in Worksheet #11, the final well locations, depths, and the screened intervals for those wells will be based on site stratigraphy, site hydrogeology, and the distribution of contaminants determined during the investigation. Groundwater samples will be collected for fixed-base laboratory analysis. Field water quality data and water level measurement data will also be collected.

A landfill gas survey will be conducted at the site to verify potential landfill gas production.

**SAP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table**  
 (UFP-QAPP Manual Section 3.1.1)

<b>SAMPLING LOCATION/ IDENTIFICATION NUMBER<sup>1</sup></b>	<b>MATRIX</b>	<b>DEPTH (units)</b>	<b>ANALYTICAL GROUP</b>	<b>NUMBER OF SAMPLES (identify field duplicates)</b>	<b>SAMPLING SOP REFERENCE<sup>3</sup></b>
02SGORE-MMMMMM*	Soil Gas	Hand auger (0 to 3 feet)	PCE, TCE, c-,t-1-2-DCE, BTEX, and TPH	49 GORE-SORBER <sup>®</sup> plus additional samples as needed	Tetra Tech SOPs SA- 1.3, CT-04, and HS-1.0
02SB01(-28)NNNN-RR <sup>2</sup>	Subsurface Soil	6 inches to 8 feet, depending on the highest PID reading <sup>4</sup>	VOCs SVOCs Pesticides/PCBs Herbicides Inorganics	10 (plus up to 8 additional samples as needed)  3 duplicates should be collected	Tetra Tech SOPs SA- 1.3, SA-2.5, SA-6.3, CT-04, and HS-1.0
02SD01(-15)NNNN-RR <sup>2</sup>	Sediments	0 to 6 inches	VOCs SVOCs Pesticides/PCBs Herbicides Inorganics Dioxins/Furans	9 (plus up to 5 additional samples as needed)  2 duplicates should be collected	Tetra Tech SOPs SA- 1.2, SA-6.3, GH-1.5, and CT-04
02SWW01(-15)-RR <sup>2</sup>	Surface Water	Grab sample collocated with the sediment sample	VOCs SVOCs Pesticides/PCBs Herbicides Inorganics	4 (plus up to 5 additional samples as needed)  2 duplicates should be collected	Tetra Tech SOP SA- 1.2, SA-6.3, CT-04, and GH-2.4
02GW01(-28)-RR <sup>2</sup>	Groundwater	Up to 40 feet	VOCs SVOCs Pesticides/PCBs Herbicides Inorganics Dioxins/Furans	10 (plus up to 8 additional samples as needed)  3 duplicates should be collected	Tetra Tech SOP SA- 1.1, SA-6.3, GH-1.2, GH-2.4, GH-2.8, and CT-04

Notes:

<sup>1</sup>The identification number consists of, in order, 02 for Site 2; SS for surface soil, SB for subsurface soil, GW for groundwater, SW for surface water, SD for sediment; and then the number of the sample, starting at 01 (up to the maximum number of samples that will be collected for each matrix). For soil samples, the depth will also need to be included at the end of the identification. For example, the first subsurface soil sample. For soil samples, the depth will also need to be included at the end of the identification. For example, a sample taken at a depth of 2 to 4 feet on the 3<sup>rd</sup> event would be 02SB010204-03. The duplicate samples will be identified by adding a D at the end of the identification number; therefore, it will be 02SB010204-03D. Further definitions can be found in SOP CT-04 included in Appendix D.

<sup>2</sup>MMMMM stands for the serial number provided by the manufacturer for each GORE-SORBER<sup>®</sup> module that will be installed. NNNN stands for the depth of the sample collected. RR stands for the event number (either 03 or 04).

<sup>3</sup>Refer to the Project Sampling SOP References Table (see Worksheet #21).

<sup>4</sup>Samples will be analyzed using a PID, and samples to be sent to the laboratory will be selected based on the PID readings. For further explanation, see Worksheet #17.

PCE = Tetrachloroethylene

TCE = Trichloroethylene

DCE = Dichloroethene

BTEX = Benzene, toluene, ethylbenzene, and xylenes

**SAP Worksheet #19 – Analytical SOP Requirements Table**  
 (UFP-QAPP Manual Section 3.1.1)

<b>MATRIX</b>	<b>ANALYTICAL GROUP</b>	<b>ANALYTICAL AND PREPARATION METHOD/ SOP REFERENCE <sup>(1)</sup></b>	<b>CONTAINERS</b> (number, size, and type)	<b>SAMPLE VOLUME</b> (units)	<b>PRESERVATION REQUIREMENTS</b> (chemical, temperature, light protected)	<b>MAXIMUM HOLDING TIME</b> (preparation/ analysis)
Groundwater, surface water, and aqueous QC blanks	VOCs	SW-846 5030/8260B, Empirical SOP-202	Three 40-milliliter (mL) glass vials	5 mL	Hydrochloric acid (HCl) to pH < 2; Cool to 4 (± 2) °C; no headspace	14 days to analysis
Soil and sediment	VOCs	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram Encore samplers or terracores	5 grams	Sodium bisulfate in water, freeze to < -10 °C	48 hours from sampling to preparation, 14 days to analysis
Groundwater, surface water, and aqueous QC blanks	SVOCs (including low level PAHs)	SW-846 3510C/3520/8270D/8270D-Low, Empirical SOP-201/300	Two 1-liter glass amber bottles	1,000 mL	Cool to 4 (± 2) °C	7 days until extraction, 40 days to analysis
Soil and sediment	SVOCs (including low level PAHs)	SW-846 3540/3550/8270D/8270D-Low, Empirical SOP-201/343	One 4-ounce glass jar	30 grams	Cool to 4 (± 2) °C	14 days until extraction, 40 days to analysis
Groundwater, surface water, and aqueous QC blanks	Pesticides	SW-846 3510C/3520/8081A, Empirical SOP-211/302	Two 1-liter glass amber bottles	1,000 mL	Cool to 4 (± 2) °C	7 days until extraction, 40 days to analysis
Soil and sediment	Pesticides	SW-846 3540/3545/3550/8081A, Empirical SOP-211/343	One 4-ounce glass jar	30 grams	Cool to 4 (± 2) °C	14 days until extraction, 40 days to analysis
Groundwater, surface water, and aqueous QC blanks	Herbicides	SW-846 8151A, Empirical SOP-304	Two 1-liter glass amber bottles	1,000 mL	Cool to 4 (± 2) °C	7 days until extraction, 40 days to analysis

<b>MATRIX</b>	<b>ANALYTICAL GROUP</b>	<b>ANALYTICAL AND PREPARATION METHOD/ SOP REFERENCE <sup>(1)</sup></b>	<b>CONTAINERS</b> (number, size, and type)	<b>SAMPLE VOLUME</b> (units)	<b>PRESERVATION REQUIREMENTS</b> (chemical, temperature, light protected)	<b>MAXIMUM HOLDING TIME</b> (preparation/ analysis)
Soil and sediment	Herbicides	SW-846 3550/8151A, Empirical SOP-310	One 4-ounce glass jar	30 grams	Cool to 4 ( $\pm$ 2) °C	14 days until extraction, 40 days to analysis
Groundwater, surface water, and aqueous QC blanks	PCBs	SW-846 3510C/3520/8082, Empirical SOP-211/302	Two 1-liter glass amber bottles	1,000 mL	Cool to 4 ( $\pm$ 2) °C	7 days until extraction, 40 days to analysis
Soil and sediment	PCBs	SW-846 3540/3545/3550/8082, Empirical SOP-211/343	One 4-ounce glass jar	30 grams	Cool to 4 ( $\pm$ 2) °C	14 days until extraction, 40 days to analysis
Groundwater, surface water, and aqueous QC blanks	Metals, Including Mercury (and Dissolved Iron and Manganese)	SW-846 3010A/6010C/7470A, Empirical SOP-100/103/105	One 500-mL plastic bottle	50 mL/ 30 mL mercury	Nitric acid to pH <2; Cool to 4 ( $\pm$ 2) °C	180 days to analysis except mercury, 28 days for mercury
Soil and sediment	Metals, Including Mercury	SW-846 3050B/6010C/7471A, Empirical SOP-100/104/105	One 4-ounce glass jar	1 to 2 grams/ 0.3 gram for mercury	Cool to 4 ( $\pm$ 2) °C	180 days to analysis except mercury, 28 days for mercury
Groundwater, surface water, and aqueous QC blanks	Cyanide	SW-846 9012A, Empirical SOP-164	One 250-mL plastic bottle	50 mL	Sodium hydroxide (NaOH) to a pH > 12; Cool to 4 ( $\pm$ 2) °C	14 days to analysis
Soil and sediment	Cyanide	SW-846 9012A, Empirical SOP-164	One 4-ounce glass jar	5 grams	Cool to 4 ( $\pm$ 2) °C	14 days to analysis
Groundwater, surface water, and aqueous QC blanks	Dioxins/Furans	SW-846 8290, APPL HPL8290	Two 1-liter glass amber bottle	1,000 mL	Cool to 4 ( $\pm$ 2) °C	30 days for extraction, 40 days for analysis

<b>MATRIX</b>	<b>ANALYTICAL GROUP</b>	<b>ANALYTICAL AND PREPARATION METHOD/ SOP REFERENCE <sup>(1)</sup></b>	<b>CONTAINERS (number, size, and type)</b>	<b>SAMPLE VOLUME (units)</b>	<b>PRESERVATION REQUIREMENTS (chemical, temperature, light protected)</b>	<b>MAXIMUM HOLDING TIME (preparation/ analysis)</b>
Soil and sediment	Dioxins/Furans	SW-846 8290, APPL HPL8290	One 8-ounce glass jar with Teflon <sup>®</sup> -lined lid	30 grams	Cool to 4 (± 2) °C	30 days for extraction, 40 days for analysis
Groundwater	Dissolved gases (methane, ethane, ethene)	RSK SOP 175, Empirical SOP-236	Three 40-mL glass vials	15 mL	HCl to pH <2; Cool to 4 (± 2) °C	14 days
Groundwater	TOC	SW-846 9060/9060A, Empirical SOP-221	One 500-mL plastic bottle	250 mL	Sulfuric acid to pH <2; Cool to 4 (± 2) °C	28 days
Groundwater	Anions (nitrate, nitrite, chloride and sulfate)	USEPA 300.0, Empirical SOP-145	One 500-mL plastic bottle	5 mL for each analyte	Cool to 4 (± 2) °C	Nitrate/Nitrite -48 hours Chloride/ Sulfate – 28 days
Groundwater	Dissolved sulfide	SM4500S F, Empirical SOP-153	One 500-mL plastic bottle	200 mL	1 mL 2 N zinc acetate with NaOH to a pH >9; Cool to 4 (± 2) °C	7 days to analysis
Groundwater	Alkalinity	SM 2320B, Empirical SOP-154	One 500-mL plastic bottle	25 mL	Cool to 4 (± 2) °C	14 days

Notes:

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table (see Worksheet #23).

**SAP Worksheet #20 – Field QC Sample Summary Table**  
 (UFP-QAPP Manual Section 3.1.1)

MATRIX	ANALYTICAL GROUP	NUMBER OF SAMPLING LOCATIONS <sup>2</sup>	NUMBER OF FIELD DUPLICATES	NUMBER OF MS/MSDS <sup>1</sup>	NUMBER OF FIELD BLANKS	NUMBER OF EQUIP. BLANKS	NUMBER OF VOA TRIP BLANKS	TOTAL NUMBER OF SAMPLES TO LAB
Subsurface Soil	VOCs, SVOCs, Pesticides, PCBs, Herbicides and Inorganics	18	2	1	1	1	1	20
Sediment	VOCs, SVOCs, Pesticides, PCBs, Herbicides, Inorganics, Dioxins/Furans	14	2	1	1	1	1	20
Surface Water	VOCs, SVOCs, Pesticides, PCBs, Herbicides, and Inorganics	9	2	1	1	1	1	15
Groundwater	VOCs, SVOCs, Pesticides, PCBs, Herbicides, Inorganics, Dioxins/Furans	18	2	1	1	1	1	24

Notes:

MS/MSD = Matrix spike/matrix spike duplicate  
 VOA = Volatile organic analysis

<sup>1</sup> Although the MS/MSD is not typically considered a field QC, it is included here because location determination is often established in the field.

<sup>2</sup> If samples are collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

**SAP Worksheet #21 – Project Sampling SOP References Table**

REFERENCE NUMBER	TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
SA-1.1	Title: Groundwater Sample Acquisition and Onsite Water Quality Testing Revision: <u>November 1, 2007</u> Number: <u>SESDPROC-209-R1</u>	Tetra Tech	NA	N	Contained in Appendix D
SA-1.2	Title: Surface Water and Sediment Sampling Revision: <u>January 28, 2008</u> Number: <u>SESDPROC-011-R2</u>	Tetra Tech	Sample log sheets, boring logs	N	Contained in Appendix D
SA-1.3	Title: Soil Sampling Revision: <u>November 1, 2007</u> Number: <u>SESDPROC-010-R3</u>	Tetra Tech	Field log book, sample log sheets, boring logs	N	Contained in Appendix D
SA-2.4	Title: <u>Soil Gas Sampling</u> Revision: <u>January 28, 2009</u> Revision 2	Tetra Tech	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate-free detergent, deionized water	N	Contained in Appendix D
SA-2.5	Title: <u>Direct Push Technology (Geoprobe®/Hydropunch™)</u> Effective Day: <u>September, 2003</u> Revision 3	Tetra Tech	Geoprobe and sampling equipment	N	Contained in Appendix D
SA-6.3	Title: <u>Field Documentation</u> Revision: <u>March 9, 2009</u> Revision 3	Tetra Tech	Log book	N	Contained in Appendix D
SA-7.1	Title: <u>Management of Investigation Derived Waste</u> Revision: <u>November 1, 2007</u> Number: <u>SESDPROC-202-R1</u>	Tetra Tech	NA	N	Contained in Appendix D
CT-04	Title: <u>Sample Nomenclature</u> Effective Day: <u>March 9, 2009</u> Revision 2	Tetra Tech	NA	N	Contained in Appendix D
CT-05	Title: <u>Database Record and Quality Assurance</u> Effective Day: <u>January 29, 2001</u> Revision 2	Tetra Tech	NA	N	Contained in Appendix D

REFERENCE NUMBER	TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
GH-1.2	Title: <u>Evaluation of Existing Monitoring Wells and Water Level Measurement</u> Effective Day: <u>September 2003</u> <u>Revision 2</u>	Tetra Tech	NA	N	Contained in Appendix D
GH-1.5	Title: <u>Borehole and Sample Logging</u> Effective Day: <u>June 1999</u> <u>Revision 1</u>	Tetra Tech	NA	N	Contained in Appendix D
GH-2.4	Title: <u>In-Situ Hydraulic Conductivity Testing</u> Effective Day: <u>June 1999</u> <u>Revision 1</u>	Tetra Tech	NA	N	Contained in Appendix D
GH-2.8	Title: <u>Groundwater Monitoring Well Installation</u> Effective Day: <u>September 2003</u> <u>Revision 3</u>	Tetra Tech	Health and safety equipment, well drilling and installation equipment, hydrogeologic equipment, drive point installation tools	N	Contained in Appendix D
GH-3.1	Title: <u>Resistivity and Electromagnetic Induction</u> Effective Day: <u>June 1999</u> <u>Revision 1</u>	Tetra Tech		N	Contained in Appendix D
GH-3.2	Title: <u>Magnetic and Metal Detection Survey</u> Effective Day: <u>June 1999</u> <u>Revision 1</u>	Tetra Tech	Metal detectors	N	Contained in Appendix D
HS-1.0	Title: <u>Utility Locating</u> Effective Day: <u>September 2003</u> <u>Revision 3</u>	Tetra Tech		N	Contained in Appendix D
DV-01	Title: <u>Data Validation- Contract Laboratory Program (CLP) Organics for Solid and Aqueous Matrices</u> Effective Day: <u>January 28, 2009</u> <u>Revision 3</u>	Tetra Tech	NA	N	Contained in Appendix D
DV-03	Title: <u>Data Validation- CLP Inorganics for Solid and Aqueous Matrices</u> Effective Day: <u>February 2, 2009</u> <u>Revision 0</u>	Tetra Tech	NA	N	Contained in Appendix D

**SAP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table**  
 (UFP-QAPP Manual Section 3.1.2.4)

FIELD EQUIPMENT	ACTIVITY <sup>1</sup>	FREQUENCY	ACCEPTANCE CRITERIA	CA	RESPONSIBLE PERSON	SOP REFERENCE <sup>2</sup>	COMMENTS
Water Quality Meter	Visual Inspection	Daily	Manufacturer's guidance	Operator correction or replacement	FOL	Manufacturer's guidance manual	None
	Calibration/ Verification	Beginning and end of day					
Turbidity Meter	Visual Inspection	Daily	Manufacturer's guidance	Operator correction or replacement	FOL	Manufacturer's guidance manual	None
	Calibration/ Verification	Beginning and end of day					
Water Level Indicator	Visual Inspection	Daily	0.01 foot accuracy	Operator correction or replacement	FOL	Manufacturer's guidance manual	None
	Field checks as per manufacturer	Once upon receiving from vendor					
PID	Visual Inspection	Daily	Manufacturer's guidance	Operator correction or replacement	FOL	Manufacturer's guidance manual	None
	Calibration/ Verification	Beginning and end of day					

Notes:

<sup>1</sup> Activities may include: calibration, verification, testing, maintenance, and/or inspection.

<sup>2</sup> Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

**SAP Worksheet #23 – Analytical SOP References Table**  
 (UFP-QAPP Manual Section 3.2.1)

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-100	Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C (Revision 19, 4/20/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/metals digestion	Natural Attenuation/Preparation	Empirical	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique Methods SW846 7470A & 245.1, CLP-M 4.1 (Revision 16, 1/28/09)	Definitive	Groundwater, surface water, and aqueous QC samples/mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique Methods SW846 7471A, 7471B, & 245.5, CLP-ILM 4.1 (Revision 17, 1/29/09)	Definitive	Soil and sediment/mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-105	Metals Analysis by Inductively Coupled Plasma (ICP) Technique Methods 200.7, SW846 6010B, SM 19 <sup>th</sup> Edition 2340B, USEPA ILMO 4.1 (Revision 15, 5/08/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/ Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Empirical	N
Empirical SOP-145	Determination of Inorganic Anions in Water by Ion Chromatography using Dionex DX-500 Ion Chromatographs with Hydroxide Eluent and Dionex Column AS18, Method 300.0 Guidance (Revision 6, 1/9/09)	Definitive	Groundwater and surface water/ anions	Dionex Ion Chromatography	Empirical	N
Empirical SOP-153	Sulfide Method 376.1 and Standard Methods SM4500S-F(19 <sup>th</sup> Edition) Titrametric, Iodine) with Sample Pretreatment to Remove Interfering Substances or to Concentrate the Sulfide (Revision 3, 5/27/09)	Definitive	Groundwater, surface water, and aqueous QC samples/sulfide	Buret	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-154	Alkalinity by USEPA Method 310.1, SM2320B (Revision 15, 5/27/09)	Definitive	Groundwater and surface water/ alkalinity	Buret/pH meter	Empirical	N
Empirical SOP-164	Distillation of Aqueous/Solid Samples for Total and Non-Amenable Cyanide Analysis Methods 335.1/335.4/Standard Methods SM4500-CN C, G 18 <sup>th</sup> and 19 <sup>th</sup> ED/(SW846) 9012A/USEPA CLP ILMO 4.1 (Revision 14, 3/9/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/cyanide digestion	Natural Attenuation/ Distillation	Empirical	N
Empirical SOP-175	Post-Distillation Analysis for Cyanide by Lachat Methods 335.4, SW846 9012A, USEPA-CLP 4.1; Addendum for USEPA CLP ILM 05.2 Aqueous/Soil/Sediment (Revision 9, 7/07/08)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/cyanide	Automated Ion Analyzer	Empirical	N
Empirical SOP-201	Gas Chromatography/ Mass Spectroscopy (GC/MS) Semivolatiles by Method 625 and SW846 Method 8270C and 8270D (Revision 18, 9/16/08)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/ SVOCs	Gas Chromatography Mass Spectrometry (GC/MS)	Empirical	N
Empirical SOP-202	GC/MS Volatiles by Method 624 and SW846 Method 8260B (Revision 21, 9/11/08)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/VOCs	GC/MS	Empirical	N
Empirical SOP-211	Gas Chromatography/Electron Capture Detector (GC/ECD) Organochlorine Pesticides/PCBs by USEPA Method 608 and SW846 Method 8081A/8082 (Revision 20, 4/27/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/PCBs	Gas Chromatography Electron Capture Detector (GC/ECD)	Empirical	N
Empirical SOP-221	TOC SM5310C, USEPA Method 415.1 and SW846 Method 9060/9060A and Lloyd Kahn Method (Revision 8, 4/28/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/TOC	TOC Analyzer	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction Using SW-846 Method 5035 for 8260B Analysis (Revision 8, 9/24/08)	Definitive	Soil and sediment/ VOCs extraction	GC/MS	Empirical	N
Empirical SOP-236	Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175 (Automated Headspace) (Revision 1, 4/28/09)	Definitive	Groundwater, surface water, and aqueous QC samples/dissolved gases	GC/MS SIM	Empirical	N
Empirical SOP-300	GC/MS Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis (Revision 17, 9/23/08)	Definitive	Groundwater, surface water, and aqueous QC samples/SVOCs extraction	Natural Attenuation/ Extraction	Empirical	N
Empirical SOP-302	Pesticide/PCBs- Aqueous Matrix Extraction for USEPA 608 and SW846 Method 8081A/8082 Using Method 3510C (Revision 16, 9/23/08)	Definitive	Groundwater, surface water, and aqueous QC samples/PCBs extraction	Natural Attenuation/ Extraction	Empirical	N
Empirical SOP-304	Herbicides Aqueous Matrix by SW846 Method 8151A (Revision 11, 9/23/08)	Definitive	Groundwater, surface water, and aqueous QC samples/herbicides	GC/ECD	Empirical	N
Empirical SOP-310	Herbicides Non-Aqueous Matrix by SW846 Method 8150B/8151A (Revision 11, 9/24/08)	Definitive	Soil and sediment/ herbicides	GC/ECD	Empirical	N
Empirical SOP-343	BNA, Pesticides/PCB, and TPH non-aqueous matrix microwave extraction Using SW-846 Method 3546 (Revision 0, 8/01/09)	Definitive	Soil and sediment/ SVOCs and PCBs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-404	Laboratory Sample Receiving Log-in and Storage SOPs (Revision 13, 6/29/09)	Definitive	Log-in	Natural Attenuation/ Log-in	Empirical	N
Empirical SOP-405	Analytical Laboratory Waste Disposal (Revision 5, 6/23/09)	Definitive	Disposal	Natural Attenuation/ Disposal	Empirical	N
Empirical SOP-410	SOPs for Laboratory Sample Storage, Secure Areas, and Sample Custody (Revision 7, 6/23/09)	Definitive	Log-in	Natural Attenuation/ Log-in	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
APPL HPL8290	Instrumental Analysis of Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) by High Resolution GC/MS (USEPA Method 8290) (Revision 4, 09/16/09)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/dioxins/furans	High Resolution GC/MS	APPL	N
APPL SOX8290S	PCDD and PCDF (USEPA Method 8290) Soxhlet Extraction of Soil/Sediment (Revision 1, 1/19/09)	Definitive	Soil and sediment/dioxins/furans extraction	Natural Attenuation/Extraction	APPL	N
APPL SEP8290	PCDD and PCDF (USEPA Method 8290) Separatory Extraction of Aqueous Samples(Revision 1, 09/23/09)	Definitive	Groundwater, surface water, and aqueous QC samples/dioxins/furans extraction	Natural Attenuation/Extraction	APPL	N
APPL 8290MAIN	High Resolution GC/MS Periodic Maintenance SOP (Revision 0, 11/19/08)	Definitive	Maintenance	Natural Attenuation/Periodic Maintenance	APPL	N
APPL SHR001	Receiving Samples (Revision 31, 11/11/09)	Definitive	Log-in	Natural Attenuation/Log-in	APPL	N
APPL SHR012	Sample Disposal and Waste Collection, Storage and Disposal (Revision 11, 6/26/09)	Definitive	Disposal	Natural Attenuation/Disposal	APPL	N

Copies of all the SOPs listed are included in Appendix E.

**SAP Worksheet #24 – Analytical Instrument Calibration Table**  
 (UFP-QAPP Manual Section 3.2.2)

INSTRUMENT	CALIBRATION PROCEDURE	FREQUENCY OF CALIBRATION	ACCEPTANCE CRITERIA	CA	PERSON RESPONSIBLE FOR CA	SOP REFERENCE <sup>(1)</sup>
GC/MS GC/MS - VOCs	Initial Calibration (ICAL) – Minimum of a 5-point calibration curve is prepared.	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification.	The average response factor (RF) for System Performance Check Compounds (SPCCs) must be $\geq 0.30$ for chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq 0.1$ for chloromethane, bromoform, and 1,1-dichloroethane. The relative standard deviation (RSD) for calibration check compounds (CCCs) for must be $\leq 30\%$ ; RSD for each analyte must be $\leq 15\%$ , or the linear least squares regression (r) must be $\geq 0.995$ .	Repeat calibration if criterion is not met.	Analyst, Department Manager	Empirical SOP-202/225
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) of all analytes must be within 75-125%.	Correct problem and verify second source standard. Reanalyze ICAL.	Analyst, Department Manager	
	Continuing Calibration Verification (CCV)	Perform one per 12-hour analysis period after tune.	The RF for SPCCs must be $\geq 0.30$ for chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq 0.1$ for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for CCCs must be $\leq 20\%$ .	Repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
GC/MS GC/MS – VOCs (continued)	Tune Verification - Bromofluorobenzene (BFB)	At the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Retune and/or clean source.	Analyst, Department Manager	
GC/MS SVOCs (including low level PAHs)	ICAL – A 6-point initial calibration for all analytes.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	The average RF for SPCCs must be $\geq 0.05$ . The RSD for CCCs must be $\leq 30\%$ ; RSD for each analyte must be $\leq 15\%$ , or $r \geq 0.995$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP-201 300/343
	ICV – Second Source	Perform after each ICAL.	The %R of all analytes must be within 75-125%. SPCC RFs $\geq 0.050$ ; CCCs $\leq 20\%D$ .	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after tune.	SPCC RFs $\geq 0.050$ ; CCCs $\leq 20\%D$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Tune Verification – decafluorotriphenylphosphine (DFTPP)	At the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Retune and/or clean source.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
GC/ECD Pesticides	ICAL – A 6-point calibration of individual pesticides, with a mid-point calibration of toxaphene and chlordane.	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The RSD for each analyte must be $\leq 20\%$ , or $r \geq 0.995$ .	Repeat ICAL if single point calibration for toxaphene, or chlordane is identified in analysis of sample; 6-point calibration run of identified compound with reanalysis of sample.	Analyst, Department Manager	Empirical SOP-211/302/329
	ICV – Second Source	Once after each ICAL.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Analyze standard at the beginning and end of sequence and every 10 samples.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	Breakdown Check (pesticides only)	Perform daily prior to sample analysis.	The degradation must be $\leq 15\%$ for both Endrin and DDT.	Column maintenance; injection port maintenance.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
GC/ECD Herbicides	ICAL - A 6-point calibration of individual herbicides.	Upon instrument receipt, major instrument change, or when the Calibration Verification does not meet criteria.	Calibration coefficient of determination ( $r^2$ ) must be $\geq$ 0.990.	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	Empirical SOP-304/310
	ICV – Second Source	Once after each ICAL.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Once after each ICAL.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
GC/ECD PCBs	ICAL - A 6-point calibration of Aroclor 1660 (1016/1260 mixture).	Instrument receipt, major instrument change, when CCV does not meet criteria.	$r^2$ must be $\geq$ 0.990. Mid-point calibration of other Aroclors.	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	Empirical SOP-211/302/329
	ICV – Second Source	Once after each ICAL.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Once after each ICAL.	The %R of all analytes must be within 80-120%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
ICP-AES - Metals	ICAL - the instrument is calibrated by a 1-point calibration per manufacturer's guidelines.	At the beginning of each day, or if the QC is out of criteria.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, $r \geq 0.995$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP-100/105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 90-110% of the true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration.	Analyst, Department Manager	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Continuing Calibration Blank	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst, Department Manager	
	Low-Level Check Standard	Daily after ICAL and before samples.	The %R must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE<sup>(1)</sup></b>
ICP-AES – Metals (continued)	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning and end of an analytical run and after each batch of 20 samples.	ICS A recoveries must be within the absolute value of the LOD; and ICS B recoveries must be within 80-120 %R of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
Mercury Analyzer Mercury	ICAL – A 5-point calibration curve is prepared.	Perform daily prior to sample analysis.	Must be $\leq 20\%$ RSD, $r \geq 0.995$	Recalibrate.	Analyst, Department Manager	Empirical SOP-103/104
	ICV – Second Source	Each analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
	Calibration Blank	One is performed at the beginning of analytical sequence, after every 10 samples, at the end of the sequence.	The target analyte concentration must be $< \text{LOD}$ .	Re-prepare and analyze all associated samples.	Analyst, Department Manager	
	CCV	Perform every 10 samples and at the end of the analytical sequence.	The %R must be within 80-120% of the true value.	Recalibrate.	Analyst, Department Manager	
	CCV (undistilled)	CCV (undistilled)-at beginning and end of each run sequence and every 10 samples.	The %R must be within 90-100% of the true value.	If the CCV (undistilled) fails high, report samples that are $< \text{LOQ}$ . Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
Lachat Cyanide	ICAL	Perform after major instrument maintenance and upon failure of second consecutive CCV.	r must be $\geq 0.995$ .	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	Empirical SOP-164/175
	ICV – Second Source	At the start of every sequence prepared fresh daily (undistilled).	The %R must be within 85-115% of the true value.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	
	ICB	After the ICV (undistilled).	Must be < LOD for the target analyte.	Determine source of contamination and restart sequence.	Analyst, Department Manager	
	CCV	Every 10 samples (undistilled).	The %R must be within 85-115% of the true value.	Determine cause for failure and rerun.	Analyst, Department Manager	
	ICV (distilled, high and low)	At the beginning of each set of 10 samples distilled.	The %R must be within 85-115% of the true value.	Determine cause for failure and redistill.	Analyst, Department Manager	
	Preparation Blank - undistilled)	At the beginning of each set of 10 samples distilled.	Must be < LOD for the target analyte.	Determine cause for failure and redistill.	Analyst, Department Manager	

INSTRUMENT	CALIBRATION PROCEDURE	FREQUENCY OF CALIBRATION	ACCEPTANCE CRITERIA	CA	PERSON RESPONSIBLE FOR CA	SOP REFERENCE (1)
High Resolution GC/MS Dioxins	Tune / Mass Resolution Check	At the beginning and the end of each 12-hour period of analysis.	Resolving power $\geq 10,000$ at $m/z=304.9842$ & $m/z=380.9760$ + 5 parts per million of expected mass. Lock-mass ion between lowest and highest masses for each descriptor and level of reference $\leq 10\%$ full-scale deflection.	Retune instrument and verify. Assess data for impact. If end resolution is less than 10,000, narrate or re-inject, as necessary.	Analyst, Department Manager	APPL HPL8290
	GC Column Performance Check (CPSM/WDM per method)	Prior to ICAL or CCV.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of $\leq 25\%$ ; <u>and</u> identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; <u>and</u> absolute retention times for switching from one homologous series to the next $\geq 10$ seconds for all components of the mixture.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No CA is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.	Analyst, Department Manager	
	Minimum of a 5-point ICAL for target analytes, lowest concentration standard at, or near, the LOQ.	Prior to sample analysis, as needed by the failure of CCV, and when a new lot is used as a standard source for CCV, IS or recovery standard solutions.	RSD $\leq 20\%$ for response factors for 17 unlabelled isomers & 9 labeled internal standards, <u>and</u> ion abundance ratios within limits specified in SOP; <u>and</u> signal to noise ratio $S/N \geq 10:1$ for target analytes.	Correct problem, then repeat ICAL.	Analyst, Department Manager	

INSTRUMENT	CALIBRATION PROCEDURE	FREQUENCY OF CALIBRATION	ACCEPTANCE CRITERIA	CA	PERSON RESPONSIBLE FOR CA	SOP REFERENCE (1)
High Resolution GC/MS Dioxins (continued)	CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios in accordance with SOP; <u>and</u> RF (unlabelled standards) $\leq 20\%D$ of average RF from ICAL; <u>and</u> RF (labeled standards) $\leq 30\%D$ of average RF from ICAL.	Correct problem, repeat CCV. If CCV fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV <u>End of Run CCV</u> : If RF (unlabeled standards) $> 20\%D$ and $\leq 25\%D$ and/or RF (labeled standards) $> 30\%D$ and $\leq 35\%D$ of the average RF from ICAL, then use mean RF from bracketing CCVs to quantitate impacted samples. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labeled), then run a new ICAL within 2 hours, and re-quantitate samples. Otherwise, reanalyze samples with positive detections.	Analyst, Department Manager	
GC/MS Dissolved Gases	ICAL – Minimum of a 5-point calibration curve is prepared.	Perform after major instrument maintenance and upon failure of second consecutive CCV.	RSD for each analyte must be $\leq 20\%$ , or r must be $\geq 0.995$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP-236

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
GC/MS Dissolved Gases (continued)	ICV – Second Source	Once after each initial calibration.	The %R of all analytes must be within 75-125% of the true value.	Correct problem and verify second source standard. Reanalyze ICAL.	Analyst, Department Manager	
	CCV	Perform one every 10 samples and at the end of analysis period.	The %R of all analytes must be within 80-120% of the true value.	Repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	
TOC Analyzer TOC	ICAL	Each analytical sequence.	Correlation coefficient of curve, r, must be $\geq 0.995$ .	Correct the problem, then repeat ICAL.	Analyst, Department Manager	Empirical SOP-221
	ICV – Second Source	Each analytical sequence.	The %R of all analytes must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
	CCV	Every 10 samples and at the end of the analytical sequence.	The %R of all analytes must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
IC Dionex Anions	ICAL – A minimum of 8 points and establish linear calibration range.	Perform after major instrument maintenance and upon failure of second consecutive CCV.	Must be $< 15\%$ RSD over linear range, or $r \geq 0.995$ .	Correct the problem, then repeat ICAL.	Analyst, Department Manager	Empirical SOP-145
	ICV – Second Source	After ICAL and each analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	

<b>INSTRUMENT</b>	<b>CALIBRATION PROCEDURE</b>	<b>FREQUENCY OF CALIBRATION</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>PERSON RESPONSIBLE FOR CA</b>	<b>SOP REFERENCE (1)</b>
IC Dionex Anions (continued)	CCV	Every 10 samples and at the end of the analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
Buret Sulfide	Standardization	Daily prior to sample analysis.	Standardized using 0.25 N sodium thiosulfate.	An acceptable titrant is compared against an independent source identified as an LCS/ICV.	Analyst, Department Manager	Empirical SOP-154
	ICV – Second Source	After ICAL and each analytical sequence.	The %R must be within 80-120% of the true value.	Recalibrate.	Analyst, Department Manager	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	
pH Meter Alkalinity	Standardization	Daily prior to sample analysis.	Standardize using pH 7 and pH 4, adjust as needed, and reread. Must be within $\pm 0.05$ pH units to proceed.	Re-standardize.	Analyst, Department Manager	Empirical SOP-154
	Buffer check	Check every 3 hours.	Must be within $\pm 0.20$ pH units.	Re-standardize and rerun samples.	Analyst, Department Manager	

Notes:

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table see Worksheet #23).

**SAP Worksheet #25 – Analytical Instrument & Equipment Maintenance, Testing, & Inspection Table**  
 (UFP-QAPP Manual Section 3.2.3)

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CA	RESPONSIBLE PERSON	SOP REFERENCE <sup>(1)</sup>
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-202/225

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CA	RESPONSIBLE PERSON	SOP REFERENCE <sup>(1)</sup>
GC/MS (continued)	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	SVOCs (including low level PAHs)	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-201/300/343
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Pesticides, Herbicides, and PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP 211/302/329

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CA	RESPONSIBLE PERSON	SOP REFERENCE <sup>(1)</sup>
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-100/105
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-103/104

<b>INSTRUMENT/ EQUIPMENT</b>	<b>MAINTENANCE ACTIVITY</b>	<b>TESTING ACTIVITY</b>	<b>INSPECTION ACTIVITY</b>	<b>FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>RESPONSIBLE PERSON</b>	<b>SOP REFERENCE<sup>(1)</sup></b>
Lachat	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, and change dispensing needle, all as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Cyanide	Tubing and rollers.	Prior to ICAL or as necessary.	Acceptable ICAL and CCV.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing CCV.	Analyst, Department Manager	Empirical SOP-164/175
GC/High Resolution Mass Spectrometry	Parameter Setup	Dioxins/ Furans	Physical check.	Initially; prior to daily calibration check.	Correct Parameters.	Reset if incorrect.	Analyst, Department Manager	APPL HPL8290
	Tune Check	Dioxins/ Furans	Conformance to instrument tuning.	Initially; prior to daily calibration check.	Compliance to ion abundance criteria.	Correct the problem and repeat tune check.	Analyst, Department Manager	
GC/Flame Ionization Detector	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Dissolved Gases	Injector liner, septa, column, column flow.	Daily and prior to each use.	Must meet ICAL and continuing calibration criteria.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-236

<b>INSTRUMENT/ EQUIPMENT</b>	<b>MAINTENANCE ACTIVITY</b>	<b>TESTING ACTIVITY</b>	<b>INSPECTION ACTIVITY</b>	<b>FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CA</b>	<b>RESPONSIBLE PERSON</b>	<b>SOP REFERENCE<sup>(1)</sup></b>
TOC Analyzer	Replace sample tubing, clean sample boat, replace syringe.	TOC	Tubing, sample boat, syringe	As needed.	Must meet ICAL and continuing calibration criteria.	Repeat maintenance activity of remove from service.	Analyst/ Laboratory Area Supervisor	Empirical SOP-221
IC Dionex	Check and clean segments weekly, clean reagent tubes monthly. Change syringes, eluent, and dispensing needle, all as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Anions	Check tubing. Verify that Chromatography is acceptable.	Prior to initial calibration or as necessary.	Must meet ICAL and continuing calibration criteria.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-145
Buret	Check buret for any cracks or chips. Rinse buret prior to each use and at the end of each day.	Sulfide	Visual inspection for cracks or chips.	Each use.	NA.	Remove from service.	Analyst, Department Manager	Empirical SOP-153
pH Meter	Keep probe wet at all times and inspect prior to use. Rinse thoroughly between uses.	Alkalinity	Visual inspection of probe.	Each use.	NA.	Remove from service.	Analyst, Department Manager	Empirical SOP-154

Notes:

<sup>1</sup> Specify the appropriate reference letter or number from the Analytical SOP References table (see Worksheet #23).

**SAP Worksheet #26 – Sample Handling System**  
 (UFP-QAPP Manual Appendix A)

**SAMPLE HANDLING SYSTEM**

<b>SAMPLE COLLECTION, PACKAGING, AND SHIPMENT</b>
Sample Collection (Personnel/Organization): FOL or designee/Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/Tetra Tech
Type of Shipment/Carrier: Federal Express
<b>SAMPLE RECEIPT AND ANALYSIS</b>
Sample Receipt (Personnel/Organization): Sample Custodians/Empirical and APPL
Sample Custody and Storage (Personnel/Organization): Sample Custodians/Empirical and APPL
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory, Dioxins Preparation Laboratory /Empirical and APPL
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory, Dioxins Laboratory/Empirical and APPL
<b>SAMPLE ARCHIVING</b>
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): NA
<b>SAMPLE DISPOSAL</b>
Personnel/Organization: Sample Custodians/Empirical and APPL

**SAP Worksheet #27 – Sample Custody Requirements Table**  
(UFP-QAPP Manual Section 3.3.3)

**27.1 SAMPLE NOMENCLATURE, SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES**

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. The forms will be filled in as completely as possible.

**27.1.1 Sample Identification**

Refer to Worksheet #18 for how the samples will be labeled. Also, refer to Worksheet #20 for how the field QA/QC samples will be labeled.

**27.1.2 Sample Collection Documentation**

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. The pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix D.

For sampling and field activities, the following types of information will be recorded in the field log as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site

- Visitor names, affiliations, arrival and departure times
- Health and safety issues including personal protective equipment

### **27.1.3 Sample Handling and Tracking System**

Following sample collection using the appropriate bottleware, samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottleware during shipment. The cooler will be secured using strapping/packaging tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4 ( $\pm$  2) °C until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure, which is provided in Appendix D.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during the RI. All forms must be filled in as completely as possible.

### **27.1.4 Sample Handling**

Sample handling requirements are described in Worksheet #26. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel, and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP (see Worksheet #21). Samples to be analyzed for VOCs will be accompanied by a VOC trip blank. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure integrity is preserved.

### **27.1.5 Sample Delivery**

Samples to be delivered to the laboratory will be made via a public courier (i.e., Federal Express). Samples will be sent to the laboratory within 24 hours of collection. Under no circumstances will sample holding times be exceeded.

### **27.1.6 Sample Custody**

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP SA-6.1 (see Appendix D).

A sample is under custody if any of the following apply:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The FOL is responsible for the care and custody of the samples collected until the samples are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix E.

### **27.1.7 Laboratory Custody**

Custody seals are supplied with all bottle orders. Custody seals are affixed to the cooler after sampling. The presence or absence of custody seals is noted on the Sample Receipt Condition Report.

Upon receipt of samples from the field, the laboratory sample management personnel will sign off on the chain-of-custody, open the sample cooler(s), verify sample integrity, and conduct a check against the chain-of-custody. If there is a discrepancy or problem (i.e., broken sample containers), the laboratory will contact the TOM or other qualified personnel and resolve the issue. Additionally, the laboratory completes a Sample Receipt Condition Report that documents visual inspection of the samples and specific parameters such as cooler temperature, holding times, and preservation. Discrepancies or changes will be documented on the Sample Receipt Condition Report.

The laboratory sample management personnel assign a unique laboratory work order number for the entire sample set listed on the chain-of-custody. The samples are then logged into the laboratory information management system and a Login Chain-of-custody Report is generated. Each sample within a work order is labeled numerically. Each container of a particular sample is uniquely identified by adding an alphabetical suffix to the sample number. The laboratory labels each sample container with a Laboratory Custody Label that will remain on the sample bottle for the duration of the laboratory sample storage. The laboratory also initiates the appropriate Internal Custody Record for the sample set. Laboratory personnel fill out the Internal Custody Records to document sample removal from and return to sample storage.

A laboratory data file is also initiated for the work order. This file includes the Login Chain-of-custody, Chain-of-custody, and Sample Receipt Condition Report. The folder also includes a Login File Sheet that summarizes the analyses for which the work order was logged. This sheet is used to track data completion.

Samples for a project may be batched or grouped together by the laboratory. A series of batched work orders is referred to as an SDG. The SDG includes samples received on a chain-of-custody, duplicate samples, and field QA/QC samples and can include samples of different media. QA/QC samples will be run at the frequency specified in the analytical methods. The SDG is given a specific identification number.

Samples are stored at the laboratory in refrigerators prior to, during, and after analysis. Refrigerators at the laboratory are constantly monitored for temperature. Proper temperatures and lighting are maintained in the refrigerators to ensure sample integrity and preservation. Samples are retained by the laboratory for a period of 90 days after the data report is mailed to the client unless otherwise specified in a client

contract. The laboratory then disposes of non-hazardous samples following certified disposal practices. Hazardous samples are either returned to the client or disposed of through a licensed broker. Documentation of disposal is maintained by the laboratory.

Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (Empirical SOP-404 [Laboratory Sample Receiving Log-In and Storage]) that is provided in Appendix E.

**SAP Worksheet #28 – Laboratory QC Samples Table**  
 (UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	VOCs					
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP-202/205					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix	No analytes > ½ LOQ, except common laboratory contaminants, that must be < LOQ.	Investigate source of contamination.  Rerun method blank prior to analysis of samples if possible.  Evaluate the samples and associated QC: if blank results are above LOQ, then report sample results that are < LOQ or > 10X the blank concentration.  Reanalyze blank and samples >LOQ and < 10X the blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	VOCs					
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP-202/205					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
MS/MSD	One per SDG or every 20 samples	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.  RPD $\leq$ 30%	CAs will not be taken for samples when recoveries are outside limits and surrogate and laboratory control sample (LCS) criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/Precision	Same as QC Acceptance Limits.
LCS/ Laboratory Control Sample Duplicate (LCSD) (not required)	One is performed for each batch of up to 20 samples	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.  RPD $\leq$ 30% (if LCSD)	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	VOCs					
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP-202/205					
<b>QC SAMPLE</b>	<b>FREQUENCY/NUMBER</b>	<b>METHOD/SOP QC ACCEPTANCE LIMITS</b>	<b>CA</b>	<b>PERSON(S) RESPONSIBLE FOR CA</b>	<b>DQI</b>	<b>MEASUREMENT PERFORMANCE CRITERIA</b>
Internal Standard (IS)	Three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs must be within $\pm 30$ seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	SVOCs (including low level PAHs)					
Analytical Method/SOP Reference	SW-846 8270C/8270C SIM Empirical SOP-201/300/343					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix	No target compounds should be > ½ the LOQ except common laboratory contaminants, which should be, no target compounds should be > the LOQ.	(1) Investigate source of contamination  (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
Surrogates	Six per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except Low Level PAHs which are provided in Appendix C).	(1) Check chromatogram for interference; if found, then flag data.  (2) If not found, then check instrument performance; if problem is found, then correct and reanalyze.  (3) If still out, then re-extract and analyze sample.  (4) If reanalysis is out, then flag data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	SVOCs (including low level PAHs)					
Analytical Method/SOP Reference	SW-846 8270C/8270C SIM Empirical SOP-201/300/343					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
LCS LCSD (not required)	One is performed for each batch of up to 20 samples	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except Low Level PAHs which are provided in Appendix E). RPD ≤30% (for LCS/LCSD)	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable, then narrate. If the LCS recoveries are high but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
IS	Six per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except Low Level PAHs which are provided in Appendix E). RPD ≤ 30%	CA will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias / Precision	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Pesticides/PCBs					
Analytical Method/ SOP Reference	SW-846 8081A/8082 Empirical SOP-211/302/329					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix	No analytes detected > ½ the LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, then report sample results which are <LOQ or > 10X the blank concentration.  Otherwise, re-prepare a blank and samples >LOQ and <10X LOQ.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
Surrogates	Two per sample: Tetrachloro-m-xylene (TCMX) 25-140 Decachlorobiphenyl (DCB) 40-135	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	No CA will be taken when one surrogate is within criteria. If surrogates recoveries are high and sample is <LOQ, then no CA is taken. If surrogates recoveries are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Pesticides/PCBs					
Analytical Method/SOP Reference	SW-846 8081A/8082 Empirical SOP-211/302/329					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
LCS LCSD (not required)	One is performed for each batch of up to 20 samples	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD $\leq$ 30% (for LCS/LCSD).	If an MS/MSD was performed and is acceptable, then narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be $\leq$ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias / Precision	Same as QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed	Results between primary and second column -RPD $\leq$ 40%.	None.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Herbicides					
Analytical Method/SOP Reference	SW-846 8151A Empirical SOP-304/310					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix	No analytes detected > ½ the LOQ.	Investigate source of contamination and evaluate the samples and associated QC: i.e. if the blank results are above the LOQ, then report sample results which are <LOQ or > 10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X the blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
Surrogates	One per sample: 2,4-Dichlorophenylacetic acid	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If surrogate recovery is high and sample is <LOQ, then no CA taken. If surrogate recovery is low, then the affected samples are re-extracted and reanalyzed.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Herbicides					
Analytical Method/SOP Reference	SW-846 8151A Empirical SOP-304/310					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
LCS LCSD (not required)	One is performed for each batch of up to 20 samples	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD $\leq$ 30% (for LCS/LCSD).	If an MS/MSD was performed and acceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be $\leq$ 30%.	Evaluate the samples and associated QC. If the LCS results are acceptable, narrate. If both the LCS and MS/MSD are unacceptable, re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed	Results between primary and second column - RPD $\leq$ 40%.	None.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Metals (including Mercury and Dissolved Iron and Manganese)					
Analytical Method / SOP Reference	SW-846 6010C/7470A/7471A Empirical SOP-105/103/104					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per digestion batch of 20 or fewer samples	No analytes detected > ½ the LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value; then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples	%R must be within 80-120% of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
Duplicate Sample	One per preparation batch of 20 or fewer samples of similar matrix	The %R must be within 80-120%. The RPD should be within ≤20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.
MS	One per 20 samples of similar matrix	The %R should be within 80-120%, if	Flag results for affected analytes	Analyst, Laboratory	Accuracy/Bias	Same as QC Acceptance

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Metals (including Mercury and Dissolved Iron and Manganese)					
Analytical Method / SOP Reference	SW-846 6010C/7470A/7471A Empirical SOP-105/103/104					
<b>QC SAMPLE</b>	<b>FREQUENCY/NUMBER</b>	<b>METHOD/SOP QC ACCEPTANCE LIMITS</b>	<b>CA</b>	<b>PERSON(S) RESPONSIBLE FOR CA</b>	<b>DQI</b>	<b>MEASUREMENT PERFORMANCE CRITERIA</b>
		sample < 4x spike added.	for all associated samples with "N".	Department Manager, and Data Validator		Limits.
Serial Dilution	One per preparatory batch with sample concentration(s) >50x LOD.	The 5-fold dilution result must agree within $\pm 10\%D$ of the original sample result if result is >50x LOD.	Perform Post Digestion Spike	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD.	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results of samples of same matrix as estimates in SDG narrative.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Cyanide					
Analytical Method/SOP Reference	SW-846 9010B/9012A Empirical SOP-164/175					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Calibration Blank	One is performed following each initial and continuing calibration and every 2 hours or every 10 analytical samples, whichever is more frequent	Target analyte concentration must be $< \frac{1}{2}$ LOQ.	Stop analysis, correct problem, and recalibrate.	Analyst, Laboratory Department Manager, and Data Validator	Contamination/Bias	Same as QC Acceptance Limits.
Preparation Blank	One is performed for each digestion batch	Target analyte concentrations must be $< \frac{1}{2}$ LOQ.	Re-prepare and reanalyze entire batch; except if concentration of analyte(s) in all associated samples is $> 10x$ the concentration in the blank.	Analyst, Laboratory Department Manager, and Data Validator	Contamination/Bias	Same as QC Acceptance Limits.
LCS	One is performed for each digestion batch	The %R must be within 85-115%.	Re-prepare and reanalyze the entire preparation batch.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Cyanide					
Analytical Method/SOP Reference	SW-846 9010B/9012A Empirical SOP-164/175					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
MS	One is performed for each digestion batch	The %R should be within 75-125%.	Flag data with an "N", unless recovery is > 4x the spike added; If the sample results exceed 4x the spike added, then spike the un-spiked aliquot of the sample at 2x the indigenous level or 2x the LOQ.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
Duplicate Sample	One is performed for each digestion batch	RPD of ≤20%, if concentration is > 5x LOQ; or within ± the LOQ, if the concentration is < 5x LOQ.	Flag data for associated samples with a "**".	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Dioxins					
Analytical Method/SOP Reference	SW-846 8290 APPL HPL8290					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected $\geq$ LOD or $\geq$ 20% of the associated regulatory limit or $\geq$ 5% of the sample result for the analyte, whichever is greater. (OCDD is considered a common laboratory contaminant and treated accordingly).	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.  "Totals" are not considered "target analytes" – no CA or flagging is necessary for "totals".	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
IS	Every field sample, standard and QC sample	%R for each IS in the original sample (prior to dilutions) must be within 25-150% per method.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater, Surface Water, Sediment, and Soil					
Analytical Group	Dioxins					
Analytical Method/SOP Reference	SW-846 8290 APPL HPL8290					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
LCS	One per sample preparation batch	%Rs must meet be between 70-130%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One MS/MSD per analytical/ preparation batch	%Rs should be between 70-130%.The RPD between MS and MSD should be $\leq$ 20%.	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	Dissolved Gases					
Analytical Method/SOP Reference	RSK SOP 175 Empirical SOP-236					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per batch of up to 20 samples	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, re-prepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
Calibration Blank	At the beginning of analytical sequence, after every 10 samples, at the end of the sequence	Analyte concentration must be $< 2x$ DL.	Correct problem, re-prepare, and reanalyze along with previous 10 samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 80-120% of the expected value.	Correct problem, re-prepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 75-125% of the expected value.  RPD $\leq$ 20%	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	TOC					
Analytical Method/ SOP Reference	SW-846 9060/9060A Empirical SOP-221					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per batch of up to 20 samples	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, re-prepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination / Bias	Same as QC Acceptance Limits.
Calibration Blank	At the beginning of analytical sequence, after every 10 samples, at the end of the sequence	Analyte concentration must be $< 2x$ DL.	Correct problem, re-prepare, and reanalyze along with previous 10 samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination / Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 80-120% of the expected value.	Correct problem, re-prepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 75-125% of the expected value.  RPD $\leq 20\%$	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	Anions					
Analytical Method/SOP Reference	USEPA 300.0 Empirical SOP-145					
<b>QC SAMPLE</b>	<b>FREQUENCY/NUMBER</b>	<b>METHOD/SOP QC ACCEPTANCE LIMITS</b>	<b>CA</b>	<b>PERSON(S) RESPONSIBLE FOR CA</b>	<b>DQI</b>	<b>MEASUREMENT PERFORMANCE CRITERIA</b>
Method Blank	One per batch of up to 20 samples	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
Calibration Blank	At the beginning of analytical sequence, after every 10 samples, at the end of the sequence	Analyte concentration must be $< 2x$ DL.	Correct problem, reprepare, and reanalyze along with previous 10 samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 85-115% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 80-120% of the expected value.  RPD $\leq 10\%$	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	Dissolved Sulfide					
Analytical Method/SOP Reference	SM4500S F Empirical SOP-153					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per preparation batch	No target analytes $\geq$ LOD.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
LCS	One per sample preparation batch	%R must be between 80-120%.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	Dissolved Sulfide					
Analytical Method/SOP Reference	SM4500S F Empirical SOP-153					
<b>QC SAMPLE</b>	<b>FREQUENCY/NUMBER</b>	<b>METHOD/SOP QC ACCEPTANCE LIMITS</b>	<b>CA</b>	<b>PERSON(S) RESPONSIBLE FOR CA</b>	<b>DQI</b>	<b>MEASUREMENT PERFORMANCE CRITERIA</b>
MS/MSD	One MS/MSD per analytical/preparation batch	%R must be between 75-125%. RPD ≤ 20%	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Matrix	Groundwater					
Analytical Group	Alkalinity					
Analytical Method/SOP Reference	SM2320B Empirical SOP-154					
QC SAMPLE	FREQUENCY/NUMBER	METHOD/SOP QC ACCEPTANCE LIMITS	CA	PERSON(S) RESPONSIBLE FOR CA	DQI	MEASUREMENT PERFORMANCE CRITERIA
Method Blank	One per batch of up to 20 samples	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
Calibration Blank	At the beginning of analytical sequence, after every 10 samples, at the end of the sequence	Analyte concentration must be $< 2x$ DL.	Correct problem, reprepare, and reanalyze along previous 10 samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 90-110% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 75-125% of the expected value.  RPD $\leq$ 20%	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

Notes: Bulk density analysis does not have corollary laboratory QC sample information.

**SAP Worksheet #29 – Project Documents and Records Table**  
 (UFP-QAPP Manual Section 3.5.1)

DOCUMENT	LOCATION MAINTAINED
<p>Sample Collection Documents and Records</p> <ul style="list-style-type: none"> <li>• Field logbook (and sampling notes)</li> <li>• Field sample forms (e.g. boring logs, sample log sheets, drilling logs, etc.)</li> <li>• Chain-of-custody records</li> <li>• Sample shipment airbills</li> <li>• Equipment calibration logs</li> <li>• Photographs</li> <li>• FTMR forms</li> <li>• SAP</li> <li>• Field sampling SOPs</li> </ul>	<p>Tetra Tech project file; results will be discussed in subject document.</p>
<p>Laboratory Documents and Records</p> <ul style="list-style-type: none"> <li>• Sample receipt/login form</li> <li>• Sample storage records</li> <li>• Sample preparation logs</li> <li>• Standard traceability logs</li> <li>• Equipment calibration logs</li> <li>• Sample analysis run logs</li> <li>• Equipment maintenance, testing, and inspection logs</li> <li>• CA forms</li> <li>• Reported results for standards, QC checks, and QC samples</li> <li>• Data completeness checklists</li> <li>• Sample storage and disposal records</li> <li>• Telephone logs</li> <li>• Extraction/cleanup records</li> <li>• Raw data</li> </ul>	<p>Tetra Tech project file; long-term data package storage at third party commercial document storage firm.</p>
<p>Other Documents</p> <ul style="list-style-type: none"> <li>• HASP</li> <li>• SAP</li> <li>• Field investigation data package</li> <li>• Project reports</li> </ul>	<p>Tetra Tech project file.</p>

**SAP Worksheet #30 – Analytical Services Table**  
 (UFP-QAPP Manual Section 3.5.2.3)

MATRIX	ANALYTICAL GROUP	SAMPLE LOCATIONS/ IDENTIFICATION NUMBERS	ANALYTICAL METHOD	DATA PACKAGE TURNAROUND TIME	LABORATORY / ORGANIZATION (name and address, contact person and telephone number)	BACKUP LABORATORY/ ORGANIZATION (name and address, contact person, and telephone number)
Groundwater, surface water, soil, and sediment	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Kim Kostzer <a href="mailto:kkostzer@empirlabs.com">kkostzer@empirlabs.com</a>  Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	NA
	SVOCs (including PAHs by SIM)		SW-846 8270C and 8270C SIM			
	Herbicides		SW-846 8151A			
	Pesticides		SW-846 8081A			
	PCBs		SW-846 8082			
	Metals (including mercury, dissolved iron, and manganese)		SW-846 6010B/7470A/747 1A			
	Cyanide		SW-846 9012A			
Groundwater, surface water, soil, and sediment	Dioxins/Furans	See Worksheet #18	SW-846 8290	21 calendar days	Cynthia Heeb Clark <a href="mailto:cclark@applinc.com">cclark@applinc.com</a>  APPL 908 North Temperance Avenue Clovis, CA 93611 (559) 275-2175	NA
Groundwater	Dissolved gases	See Worksheet #18	RSK SOP 175	21 calendar days	Kim Kostzer <a href="mailto:kkostzer@empirlabs.com">kkostzer@empirlabs.com</a>  Empirical 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	NA
	TOC		SW-846 9060/9060A			
	Anions (chloride, sulfate, nitrate, and nitrite)		USEPA 300.0			
	Dissolved sulfide		SM 4500S F			
	Alkalinity		SM 2320B			

**SAP Worksheet #31 – Planned Project Assessments Table**  
 (UFP-QAPP Manual Section 4.1.1)

<b>ASSESSMENT TYPE</b>	<b>FREQUENCY</b>	<b>INTERNAL OR EXTERNAL</b>	<b>ORGANIZATION PERFORMING ASSESSMENT</b>	<b>PERSON(S) RESPONSIBLE FOR PERFORMING ASSESSMENT</b> (title and organizational affiliation)	<b>PERSON(S) RESPONSIBLE FOR RESPONDING TO ASSESSMENT FINDINGS</b> (title and organizational affiliation)	<b>PERSON(S) RESPONSIBLE FOR IDENTIFYING AND IMPLEMENTING CA</b> (title and organizational affiliation)	<b>PERSON(S) RESPONSIBLE FOR MONITORING EFFECTIVENESS OF CA</b> (title and organizational affiliation)
Field Systems Audit	1 per contract year	Internal	Tetra Tech	Person assigned by Tetra Tech QAM	TOM and FOL, Tetra Tech	Auditor and TOM, Tetra Tech	CLEAN QAM, Tetra Tech
Laboratory System Audit <sup>1</sup>	3 years	External	DoD Environmental Laboratory Accreditation Program (ELAP)	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical and APPL	Laboratory QAM or Laboratory Manager, Empirical and APPL	Laboratory QAM or Laboratory Manager, Empirical and APPL

<sup>1</sup> Empirical and APPL have successfully completed the laboratory evaluation process required as part of the DoD QSM. The DoD ELAP accreditation letter is included in Appendix E.

**SAP Worksheet #32 – Assessment Findings and CA Responses**  
 (UFP-QAPP Manual Section 4.1.2)

ASSESSMENT TYPE	NATURE OF DEFICIENCIES DOCUMENTATION	INDIVIDUAL(S) NOTIFIED OF FINDINGS (name, title, organization)	TIMEFRAME OF NOTIFICATION	NATURE OF CA RESPONSE DOCUMENTATION	INDIVIDUAL(S) RECEIVING CA RESPONSE (name, title, organization)	TIMEFRAME FOR RESPONSE
Field Sampling System Audit <sup>(1)</sup>	Audit checklist (as per Navy Installation Restoration Chemical Data Quality Manual and written audit report)	Gregory Roof, TOM, Tetra Tech  William Olson, FOL, Tetra Tech  Debra Humbert, Program Manager, Tetra Tech  Mark Perry, Deputy Program Manager, Tetra Tech	Dependent on the finding; if major a stop work may be issued immediately; however, if minor within 1 week of audit	Written memo	Gregory Roof, TOM, Tetra Tech  William Olson, FOL, Tetra Tech  Debra Humbert, Program Manager, Tetra Tech  Chris Pike, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Laboratory QAM	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

Notes:

<sup>1</sup> Audits are scheduled at the Tetra Tech program level and may or may not include this project.

**SAP Worksheet #33 – QA Management Reports Table**  
 (UFP QAPP Manual Section 4.2)

<b>TYPE OF REPORT</b>	<b>FREQUENCY</b> (daily, weekly monthly, quarterly, annually, etc.)	<b>PROJECTED DELIVERY DATE(S)</b>	<b>PERSON(S) RESPONSIBLE FOR REPORT PREPARATION</b> (title and organizational affiliation)	<b>REPORT RECIPIENT(S)</b> (title and organizational affiliation)
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data	DVM or designee, Tetra Tech	TOM and project file, Tetra Tech
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem	CLEAN QAM, Tetra Tech	TOM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Project monthly progress report	Monthly for duration of the project	Monthly	Tetra Tech TOM, Tetra Tech	TOM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling is occurring	Tetra Tech FOL, Tetra Tech	TOM, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem	Laboratory PM, Empirical and APPL	TOM and project file, Tetra Tech

**SAP Worksheet #34 – Verification (Step I) Process Table**  
 (UFP-QAPP Manual Section 5.2.1)

VERIFICATION INPUT	DESCRIPTION	INTERNAL/ EXTERNAL	RESPONSIBLE FOR VERIFICATION (name, organization)
Chain-of-custody forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech TOM, and the Tetra Tech Data Validators. See SOP SA-6.3.	Internal	Sampler and FOL, Tetra Tech
SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample log sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	FOL or designee, Tetra Tech
Field QC samples	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
Chain-of-custody forms	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 – Laboratory Sample Custodian, Empirical and APPL 2 - Data Validators, Tetra Tech
Analytical data package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package.	Internal	QAM, Empirical and APPL
Analytical data package	The data package will be verified for completeness by Tetra Tech Data Validators. Missing information will be requested from the laboratory and validation will be suspended until missing data are received.	External	Data Validators, Tetra Tech
Electronic data deliverables	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness.	External	Data Validators, Tetra Tech,

Notes:

Verification includes field data verification and laboratory data verification. Verification inputs as per Worksheet #34 will be checked.

**SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table**  
 (UFP-QAPP Manual Section 5.2.2) (Figure 37, page 110 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

STEP IIa / IIb	VALIDATION INPUT	DESCRIPTION	RESPONSIBLE FOR VALIDATION (name, organization)
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Tetra Tech Project Chemist or data validators
IIa	Holding Times	Review that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Tetra Tech Project Chemist or data validators
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the measurement performance criteria listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tetra Tech Project Chemist or data validators
IIa/IIb	Field and Laboratory Duplicate Analyses for Precision	Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSD; and LCS/LCSD. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheets #12 and #28.	Tetra Tech Project Chemist or data validators
IIa/IIb	Sample Results for Representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	Tetra Tech Project Chemist or data validators
IIa/IIb	PALs	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech TOM.	Tetra Tech Project Chemist or data validators

<b>STEP IIa / IIb</b>	<b>VALIDATION INPUT</b>	<b>DESCRIPTION</b>	<b>RESPONSIBLE FOR VALIDATION (name, organization)</b>
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project data base qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPC and determine the impact of any deviations on the technical usability of the data.	Tetra Tech Project Chemist or data validators
IIa/IIb	SAP QC Sample Documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall contact the Tetra Tech TOM.	Tetra Tech TOM or designee
IIa/IIb	Documentation of Analytical Reports for Completeness	Review the chain-of-custody form generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language database.	Tetra Tech Project Chemist or data validators
IIa/IIb	PALs	Review and add project action limits to the laboratory electronic data deliverable. Flag samples and notify PM of samples that exceed PALs as listed on Worksheet #15.	Tetra Tech TOM or designee
IIb	Project Quantitation Limits for Sensitivity	Ensure that the project LOQs listed in Worksheet #15 were achieved.	Tetra Tech Project Chemist or Data Validators
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods, SOPs requirements, and matrix interferences effect on the analytical results.	Tetra Tech Project Chemist or Data Validators

**SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table**  
 (UFP-QAPP Manual Section 5.2.2.1) (Figure 37, page 110 UFP-QAPP Manual)

<b>STEP IIa / IIb</b>	<b>MATRIX</b>	<b>ANALYTICAL GROUP</b>	<b>VALIDATION CRITERIA</b>	<b>DATA VALIDATOR</b> (title and organizational affiliation)
IIa and IIb	Groundwater, surface water, soil and sediment	VOCs, dissolved gases, SVOCs/PAHs, pesticides, herbicides, and PCBs	SW-846 8260B, RSK SOP 175, 8270C, 8270C SIM, 8081A, 8151A, and 8082, method specific criteria, DoD QSM, and those criteria listed in Worksheets #12, #15, #24, and #28 will be used. If not included in Worksheet #12, #15, #24, or #28, the logic outlined in USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review USEPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater, surface water, soil and sediment	Metals and cyanide	SW-846 6010B, 7470A/7471A, and 9012A method specific criteria, DoD QSM, and those listed in Worksheets #12, #15, #24, and #28 will be used. If not included in Worksheet #12, #15, #24, or #28, the logic outlined in USEPA CLP National Functional Guidelines for Inorganic Data Review USEPA 540-R-04-004, October 2004 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater, surface water, soil, and sediment	Dioxins and furans	SW-846 8290 method specific criteria, DoD QSM, and those criteria listed in Worksheets #12, #15, #24, and #28 will be used. If not included in Worksheet #12, #15, #24, or #28, the logic outlined in USEPA CLP National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation, September 2005 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater	Anions, alkalinity, TOC, sulfide	Method-specific criteria listed in Worksheets #12, #15, #24, and #28 will be used.	Data Validation Specialist, Tetra Tech

Notes:

Bulk density data will not be validated.

**SAP Worksheet #37 – Usability Assessment**  
(UFP-QAPP Manual Section 5.2.3)

**DATA USABILITY ASSESSMENT**

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of the following DQI characteristics:

**Completeness**

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech TOM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If deviations may comprise the objectives, the Tetra Tech TOM will consult with the Navy RPM and other Project Team members, as necessary (determined by the Navy RPM), to develop appropriate CAs.

**Precision**

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

**Accuracy**

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, MS, and LCSs. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

## DATA USABILITY ASSESSMENT

### **Representativeness**

A project scientist identified by the Tetra Tech TOM and acting on behalf of the Project Team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

### **Comparability**

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Tetra Tech Project Chemist indicates that such quantitative analysis is required.

### **Sensitivity**

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

### **Project Assumptions and Data Outliers**

The Tetra Tech TOM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested.

**Describe the evaluative procedures used to assess overall measurement error associated with the project:**

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members identified by the PM will assess whether the data collectively support the attainment of project objectives. The Project Team will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use the data in a weight of evidence argument, especially when the missing or rejected data supplement other data that have not been rejected. If rejected data are used, the use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

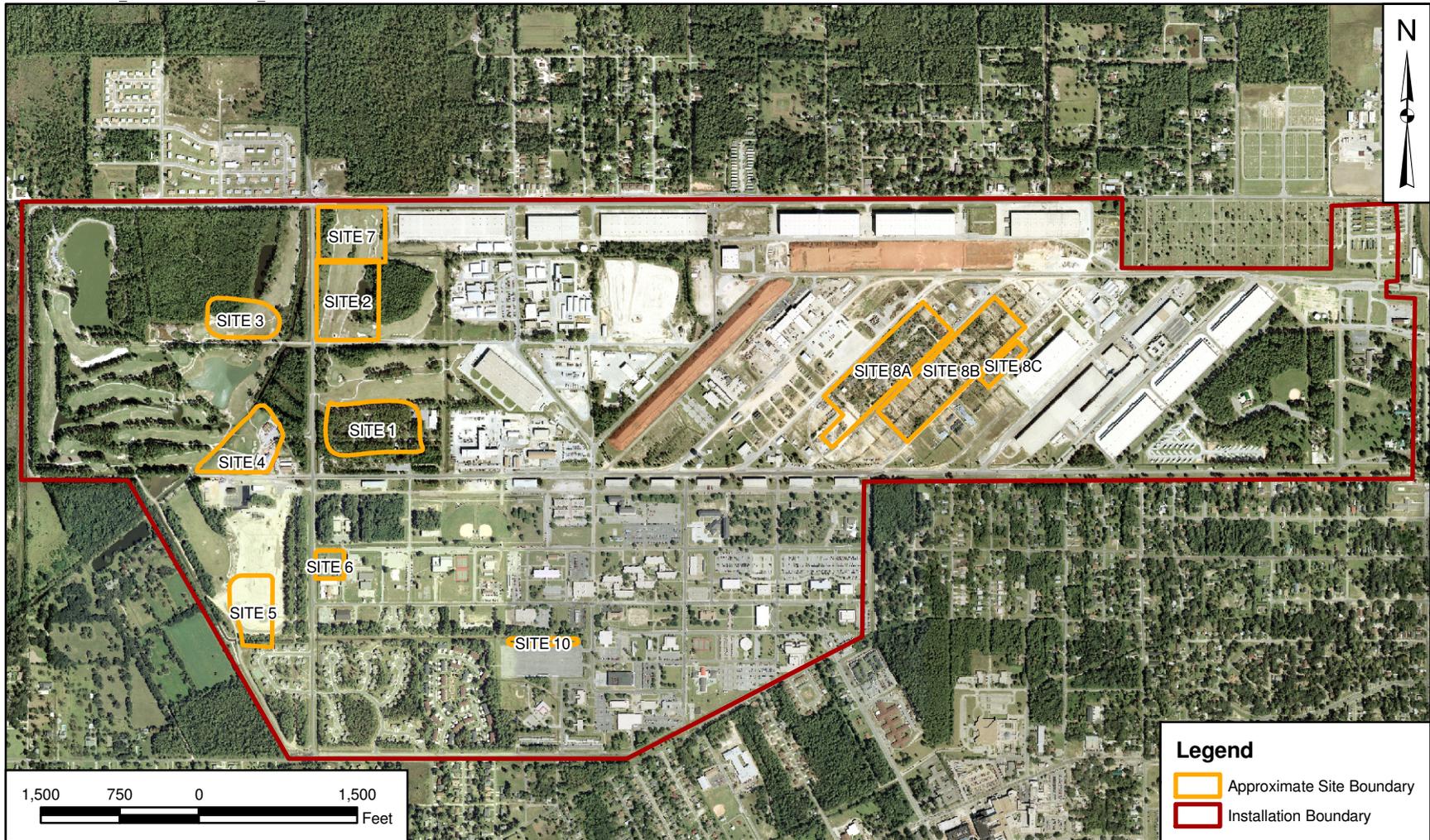
**Identify the personnel responsible for performing the usability assessment:**

The Tetra Tech TOM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, Tetra Tech TOM, the USEPA RPM, and the MDEQ RPM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face-to-face meeting or in a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

**Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:**

The data will be presented in tabular format including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other CAs, if necessary.

## FIGURES



**Legend**

- Approximate Site Boundary
- Installation Boundary

DRAWN BY K. MOORE	DATE 5/6/09
CHECKED BY Y. MARTINEZ	DATE 4/15/10
COST SCHEDULE AREA	
SCALE AS NOTED	



FACILITY MAP  
NCBC GULFPORT  
GULFPORT, MISSISSIPPI

CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 1	REV 0



**Legend**

 Approximate Site 2 Boundary

DRAWN BY K. MOORE	DATE 05/06/09
CHECKED BY P. CHURCHILL	DATE 04/01/10
COST SCHEDULE AREA	
SCALE AS NOTED	



LOCATION MAP  
 SITE 2 - WORLD WAR II LANDFILL  
 NCBC GULFPORT  
 GULFPORT, MISSISSIPPI

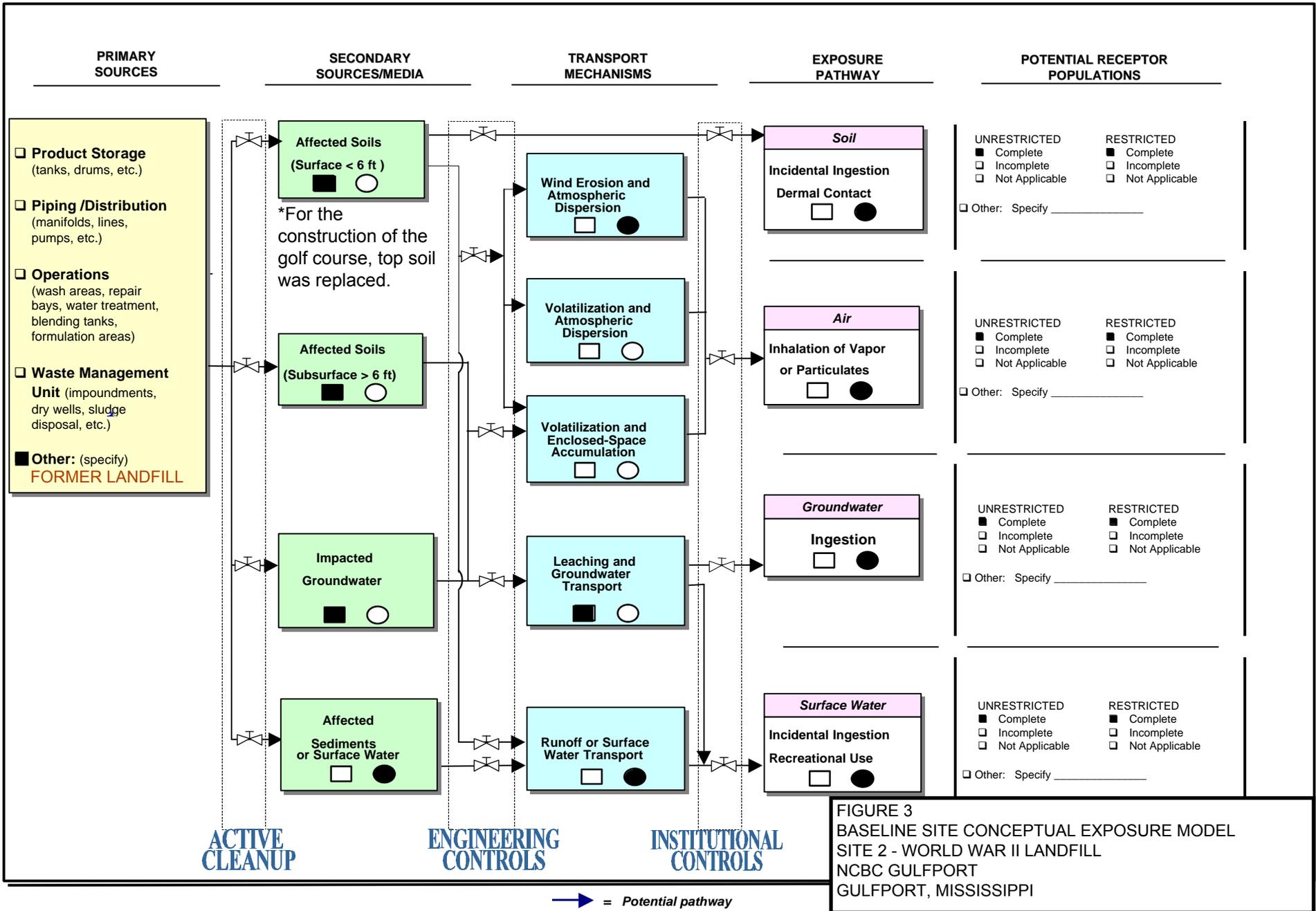
CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 2	REV 0

Site Name: **Site 2, World War II Landfill**  
 Site Location: **NCBC Gulfport, Mississippi**

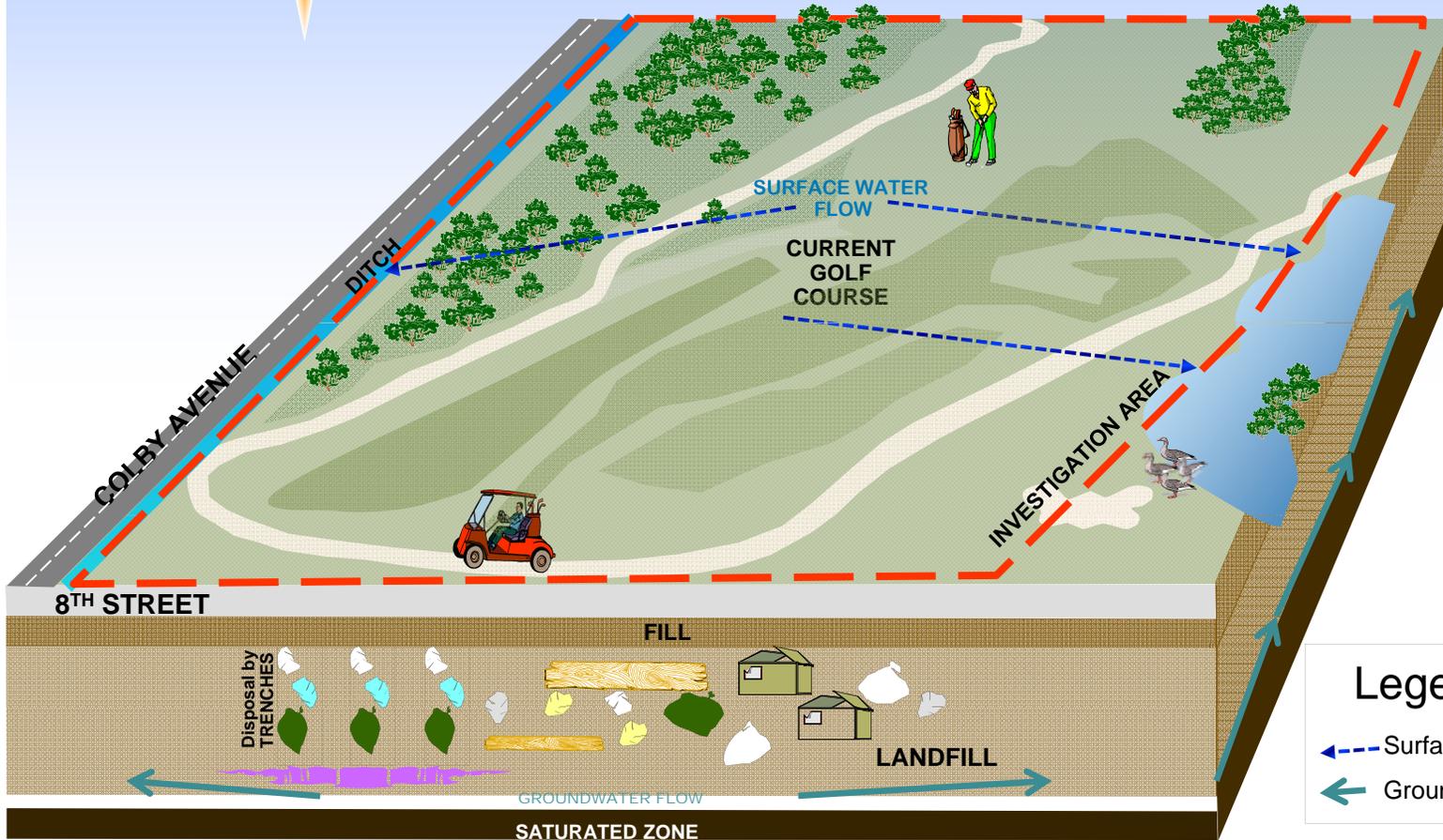
Completed By: **YML**  
 Revision Date: **5/13/2009**

Complete  
 Potentially Complete

Draft  
 Final



Generic Conceptual Site Model  
 Site 2 – World War II Landfill  
 NCBC Gulfport  
 Gulfport, Mississippi



**Legend**

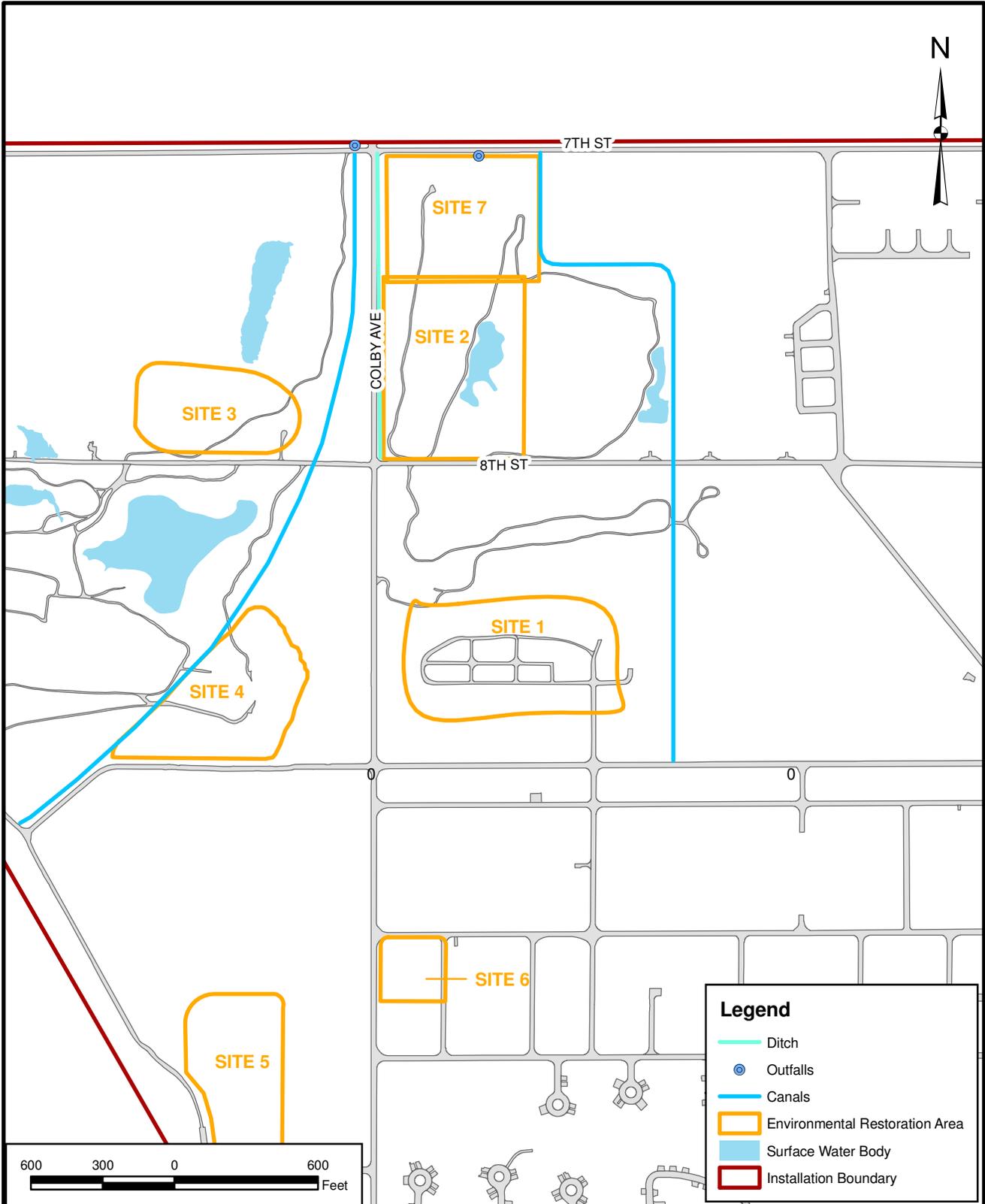
- Surface Water flow
- Groundwater flow

DRAWN BY C. Pennington	DATE 2/12/2010
CHECKED BY Y. Martinez	DATE 2/12/2010
REVISED BY	DATE
SCALE AS NOTED	



**CONCEPTUAL SITE MODEL**  
**SITE 2 - WORLD WAR II LANDFILL**  
**NCBC GULFPORT**  
**GULFPORT, MISSISSIPPI**

CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. FIGURE 4	REV 0

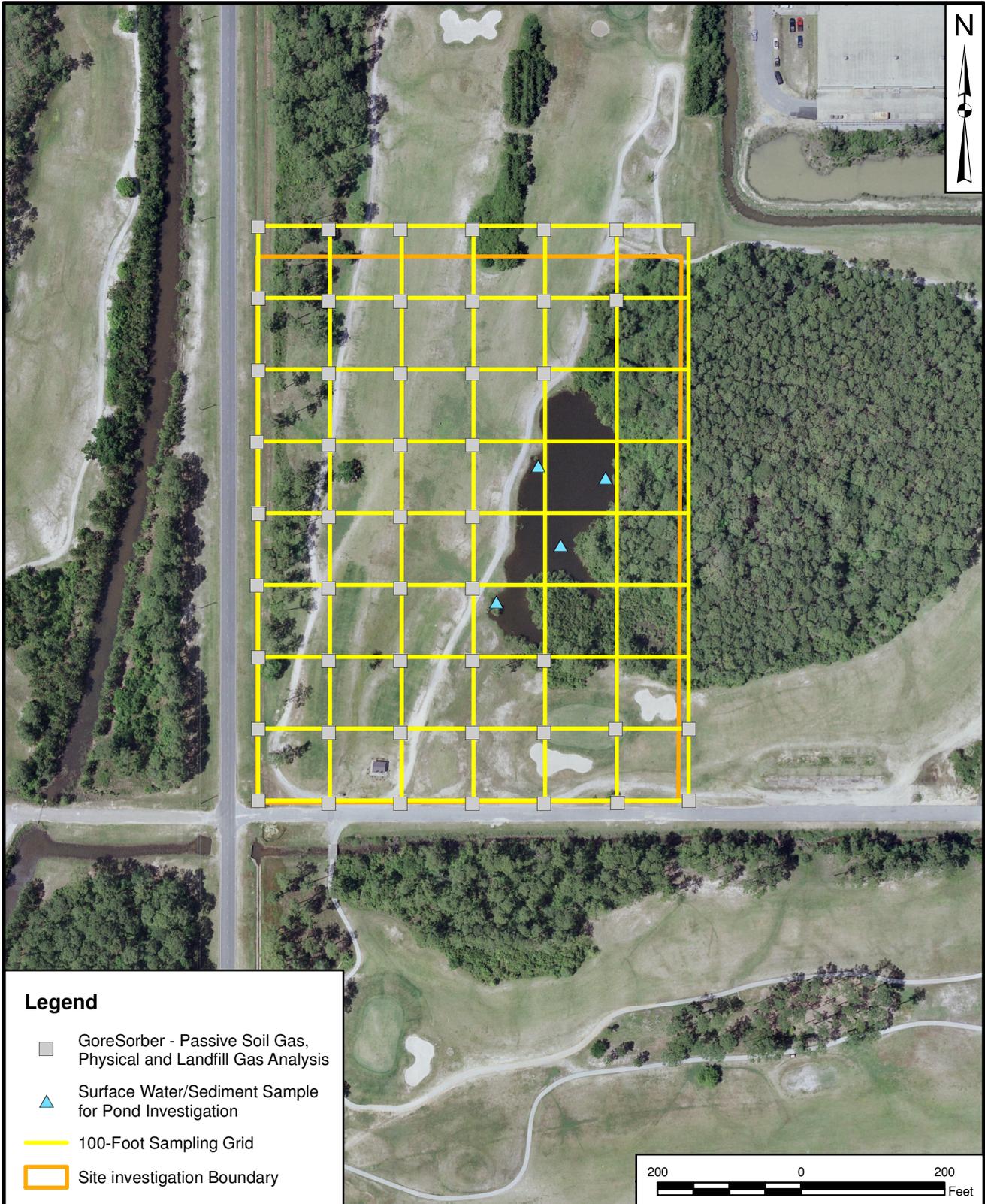


DRAWN BY S. STROZ	DATE 12/14/09
CHECKED BY Y. MARTINEZ	DATE 4/15/10
REVISED BY	DATE
SCALE AS NOTED	



**SURFACE WATER FLOW**  
**SITE 2 - WORLD WAR II LANDFILL**  
**NCBC GULFPORT**  
**GULFPORT, MISSISSIPPI**

CONTRACT NUMBER	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 5	REV 0



**Legend**

- 
 GoreSorber - Passive Soil Gas, Physical and Landfill Gas Analysis
- 
 Surface Water/Sediment Sample for Pond Investigation
- 
 100-Foot Sampling Grid
- 
 Site investigation Boundary

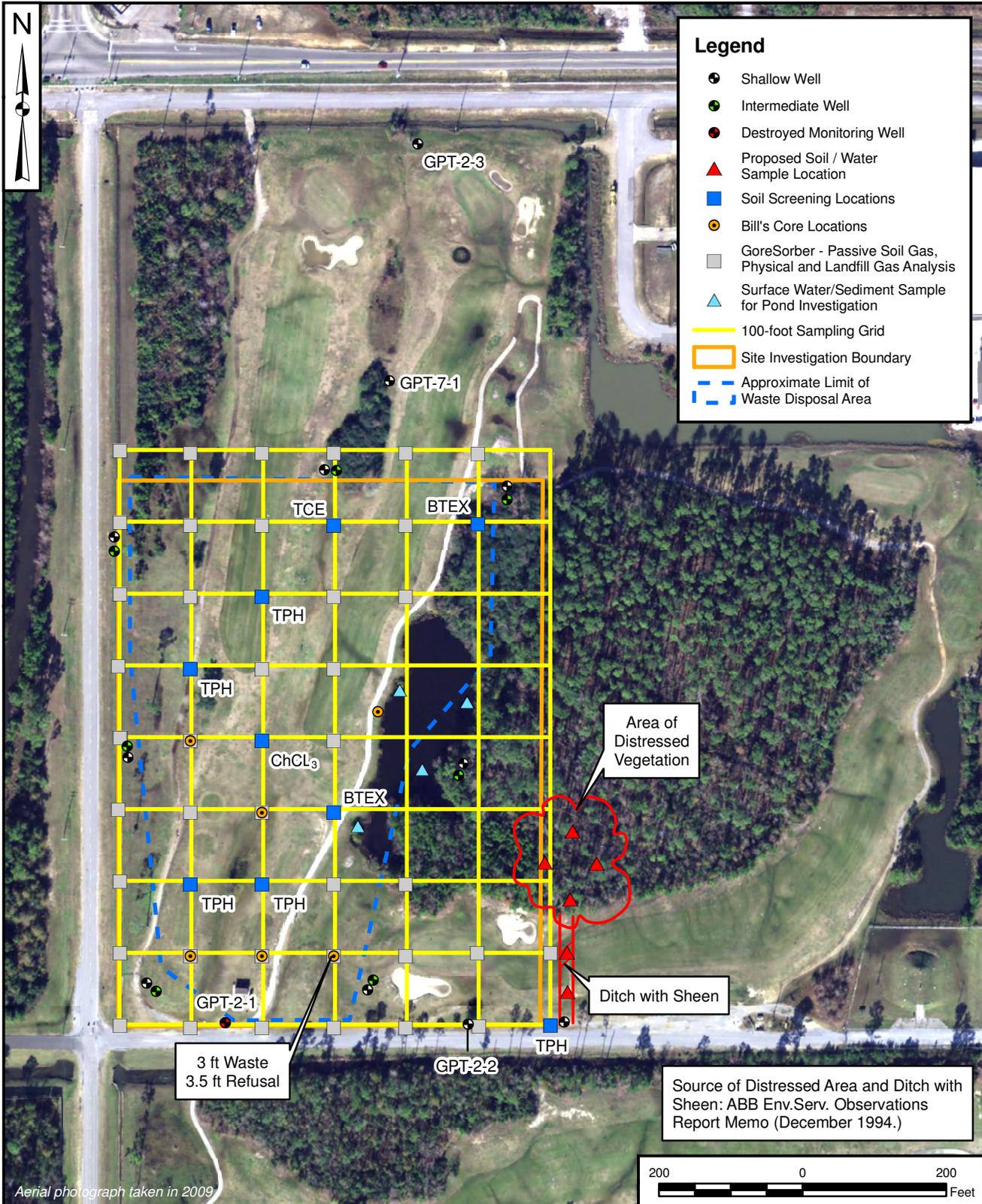


DRAWN BY S. STROZ	DATE 03/26/10
CHECKED BY Y. MARTINEZ	DATE 4/15/10
REVISED BY	DATE
SCALE AS NOTED	



**PROPOSED 100-FOOT SAMPLING GRID**  
**SITE 2 - WORLD WAR II LANDFILL**  
**NCBC GULFPORT**  
**GULFPORT, MISSISSIPPI**

CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 6	REV 0



- Legend**
- Shallow Well
  - Intermediate Well
  - Destroyed Monitoring Well
  - ▲ Proposed Soil / Water Sample Location
  - Soil Screening Locations
  - Bill's Core Locations
  - GoreSorber - Passive Soil Gas, Physical and Landfill Gas Analysis
  - ▲ Surface Water/Sediment Sample for Pond Investigation
  - 100-foot Sampling Grid
  - Site Investigation Boundary
  - - - Approximate Limit of Waste Disposal Area

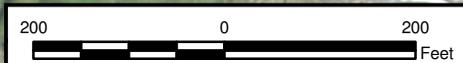
Area of Distressed Vegetation

Ditch with Sheen

3 ft Waste  
3.5 ft Refusal

Source of Distressed Area and Ditch with Sheen: ABB Env.Serv. Observations Report Memo (December 1994.)

Aerial photograph taken in 2009



DRAWN BY J. ENGLISH	DATE 06/20/11
CHECKED BY B. MARSHALL	DATE 06/24/11
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED 100-FOOT SAMPLING GRID  
SITE 2 - WORLD WAR II LANDFILL  
NCBC GULFPORT  
GULFPORT, MISSISSIPPI

CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 6A	REV 0

## REFERENCES

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ABB, 1995. Letter Report: Base-wide Groundwater, Surface Water, and Sediment Sampling Field Program and Analytical Results; Naval Construction Battalion Center, Gulfport, Mississippi. From Ms. Penny Baxter (ABB) to Mr. Art Conrad (Southern Division Naval Facilities Engineering Command). March 24, 1995.

Agent Orange/Dioxin Committee, 2002. The History Of Agent Orange Use In Vietnam. United States - Vietnam Scientific Conference on Human Health And Environmental Effects Of Agent Orange/Dioxins. March 3-6, 2002 Hanoi, Vietnam.

Envirodyne Engineers, Inc., 1985. *Initial Assessment Study*, Naval Construction Battalion Center, Gulfport, Mississippi. July.

Harding Lawson Associates, 1988. *Final Verification Report*, Naval Construction Battalion Center, Gulfport, Mississippi. July.

Harding Lawson Associates, 1999. Groundwater Monitoring Report Naval Construction Battalion Center Gulfport, Mississippi Unit Identification No.: N62604 Contract No.: N62467-89-D-0317/150 Prepared by: Harding Lawson Associates December 1999

Mississippi Department of Environmental Quality, 2002. *Risk Evaluation Procedures for Voluntary Cleanup and Redevelopment of Brownfield Sites*, Tier I Evaluation Target Risk Level. February

Petry, D. E. and R. E. Switzer, 2001. *Arsenic Concentrations in Selected Soils and Parent Materials in Mississippi*. Mississippi State University, Division of Agriculture, Forestry, and Veterinary Medicine, Office of Agricultural Communications. MAFES Bulletin 1104, June.

Suter, G. W, II, and C. L. Tsao, 1996. *Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects on Aquatic Biota: 1996 Revision*. Oak Ridge National Laboratory, ES/ER/TM-96/R2, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Tetra Tech, 2009. *Health and Safety Plan for Remedial Investigation/Feasibility Study for Site 2*, NCBC Gulfport, Mississippi. June.

## REFERENCES (CONTINUED)

USEPA (United States Environmental Protection Agency), 1989. *Risk Assessment Guidance for Superfund: Volume I, Human Health Evaluation Manual (Interim Final)*. USEPA/540/1-89/002, Washington, D.C. December.

USEPA, 1993. *Presumptive Remedy for CERCLA Municipal Landfill Sites. Office Emergency and Remedial Response Hazardous Site Control Division 5203G Quick Reference Fact Sheet*, Directive: 9355.0-49FS; USEPA 540-F-93-035; PB 93-963339. September.

USEPA, 1996a. *Soil Screening Guidance for Chemicals*, Generic Soil Screening Levels for the Inhalation of Volatiles and Fugitive Dusts. Calculated online at [http://risk.lsd.ornl.gov/calc\\_start.shtml](http://risk.lsd.ornl.gov/calc_start.shtml).

USEPA, 1996b. *Application of the CERCLA Municipal Landfill Presumptive Remedy to Military Landfills*. United States USEPA Office of Solid Waste and Emergency Response. Washington, D.C. OSWER Directive 9355.067-FS USEPA/540/F-96/020. December.

USEPA, 2001. *Ecological Screening Values*. USEPA Region 4. November.

USEPA, 2002. *Guidance for Quality Assurance Project Plans, QA/G-5, QAMS*. Office of Environmental Information, Washington, DC. December.

USEPA, 2003. *RCRA Soil Ecological Screening Levels*. USEPA Region 5. August.

USEPA, 2005. *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)*, Evaluating, Assessing, and Documental Environmental Data Collection and Use Programs. Part 1: UFP-QAPP Manual. Final Version 1. USEPA: USEPA-505-B-04-900A; DoD: DTIC ADA 427785.

USEPA, 2006a. *Freshwater Screening Benchmarks*. USEPA Region 3 BTAG. July.

USEPA 2006b. *Freshwater Sediment Ecological Screening Benchmarks*. USEPA Region 3 BTAG. August.

USEPA, 2009a. *Regional Screening Levels for Chemical Contaminants at Superfund Sites*, USEPA Regions 3, 6, and 9. May.

## REFERENCES (CONTINUED)

USEPA, 2009b. *National Primary Drinking Water Regulations, Maximum Contamination Levels*. EPA 816-F-09-004. May.

USEPA, 2009c. *Soil Screening Level, Migration to Groundwater*. USEPA Regions 3, 6, and 9. May.

**APPENDIX A**

**DQO MEETING MINUTES**

**MINUTES**  
NCBC Gulfport Tier I Meeting  
Biloxi, Mississippi

**May 12 – 13, 2009**

**Meeting Attendees**

**Team Members:**

Gordon Crane	NCBC Gulfport, IRP Manager
Bob Merrill	MDEQ, State RPM
Robert Fisher	NAVY RPM
Nancy Rouse	The Management Edge, Facilitator
Yarissa Martínez	Tetra Tech NUS, Inc., Project Manager
Jacqueline Strobl	Tetra Tech NUS, Inc., Scribe
John Overholter	CH2M Hill, Project Manager
Helen Lockard	NAVY, Tier II Link
Peggy Churchill -2 <sup>nd</sup> day	TtNUS, DQO Facilitator

**1. 1<sup>st</sup> Day Check-In (Tuesday, May 12, 2009)**

Welcome and Administrative – Nancy Rouse

- Proxies/Guests – There were no proxies. Guest Helen Lockard (Tier II Link).
- Review Agenda – adjustments noted.

Each meeting attendee provided a brief personal update. The agenda was reviewed and adjusted as necessary. Team Ground Rules have not been finalized; therefore its review was postponed and will instead be discussed during the partnering exercise.

*Parking Lot - The Action Items will be reviewed / projected after the break.*

**2. Tier II Update – Helen Lockard**

Helen Lockard provided a Tier II update to the Team, noting the recent induction of new members and the decision to have the same Management Edge facilitator, Nancy Rouse, for all the Mississippi Tier I teams. Helen noted that she would be serving as the Tier II Link for the Gulfport Partnering Team; Debbie Humbert will serve as the Tier II Link alternate.

Mrs. Lockard explained that Tier II attempts to select a non-biased, non-involved member to serve as a link in order to communicate between Tier I and Tier II teams. Helen noted that the Tier II Links are not considered to be voting members; the intention is to help and observe rather than actively participate. The Tier II Link is supposed to be objective, which is easier if you are not heavily involved. The intention is to provide information on global policy updates, DOD

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changes, and offer assistance concerning any issues that may not be able to be resolved at the Tier I level.

Mrs. Lockard noted that discussion concerning the possibility of a joint Tier I/II meeting was on the agenda. It was stated that there was potential for training, technical presentations, as well as discussions/presentations on what other teams are doing.

The other item discussed/stressed at the last Tier II meeting concerning Gulfport was the updated exit strategy. Tier II hasn't seen an exit strategy for Gulfport in a very long time. The exit strategy is supposed to be discussed during the Tier II meetings.

Mrs. Lockard briefly discussed the UFP-SAP and IROD documents. The UFP-SAP, which will be used for all sampling, is a format that attempts to standardize everything. The IROD guidance has been unclear; the Navy is currently working with TtNUS to work on streamlining the document format in order to avoid redundant information and establish consistency. Helen also stated that within each format there is enough flexibility to tailor them to fit each state.

Mr. Fisher brought up the possibility of questions/discussion RAC IV/CLEAN IV and suggested adding it to the agenda. The team expressed interest in exploring the topic.

### **3. RAB Meeting – Yarissa Martinez**

The Team briefly discussed the RAB Meeting held the previous night. The new location, though a bit difficult to find, was well received. Unfortunately there was a very low turn out.

During the RAB Meeting Marie Hansen (RAB Member) brought up questions concerning AOC 4. Mr. Fisher pointed out the area in question on a figure for the Team and provided a brief overview concerning the AOCs' history and MDEQ requests.

Bob Merrill stated that MDEQ doesn't need the Navy to sample out there at this time, but would prefer to have an EM survey conducted, because of the community's concern.

Mr. Fisher noted that the TtNUS field crew was out collecting samples for Phase II on the Off-Base AOCs. MDEQ is interested in collecting split sampling, but it was not possible at this time.

Mr. Fisher resumed discussion concerning Marie Hansen's AOC 4 questions, noting that he had reported back to her several RABs ago. MDEQ definition of AOC 4 could not be confirmed at the field. However, the drainage through that entire area flows towards Turkey Creek. Therefore, if there is something in the drainage, it will end up in Turkey Creek and will show up when the sediment is sampled. During the first phase, nothing was found that could indicate that there is a problem. During the Phase II, this will be investigated further, but if no evidence is found to support the claim of the community, this would be the last investigation phase for the AOCs. The Navy has investigated several of the community's stories and only one led to finding anything.

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Mr. Fisher stated that unless something shows up north of the landfill, then the investigation should be considered complete. Mrs. Lockard noted that this has not been discussed at the Tier II level. It has been a matter discussed internally by the Navy lawyers.

Merrill stated that the only way the Navy could be held responsible for any drum burial or disposal in the off-base areas would be if drums, still intact and recognizable as Herbicide-Orange drums, were found.

*Parking lot – Next steps of investigation.*

If the investigation results are inconclusive, the next step would be more of a legal matter, since it is not likely that funds could be used based on speculation. Further guidance would be necessary.

Navy noted that once the data was received from the samples collected this week, we should be able to conclude that there is no reason to continue the investigation.

**4. Review of Previous Action Items**

The Team took a short break while TtNUS set the projector. The Team reviewed the Ongoing Action Items:

<b>Ongoing Action Items</b>					
<b>Action Item No.</b>	<b>Responsible Party</b>	<b>Status</b>	<b>Due Date</b>	<b>Action Item</b>	<b>Comments</b>
A-1108-01	N. Rouse	Ongoing	1/22/09	Develop cost proposal for adding RAB members.	
A-1108-03	B. Fisher	Completed	5/12/09	Summarize status of off-base property issues in an e-mail to the team.	Bob will report to the team at the next Tier I meeting.
A-1108-04	G. Crane & B. Fisher	Ongoing	TBD	Track resolution of 8B and 8C wear surface and keep team apprised.	Milcon project, in various phase (not IR money).
A-1108-05	B. Fisher	Ongoing	TBD	Contract development of a monitoring plan for Site 8.	Funding has been allocated for the monitoring plan and will be contracted. Working on defining the LTM process with MDEQ.
A-0209-01	G. Crane	Completed		Will get more information from Tier II on “Joint Meeting”	The Tier II link will provide more information at the next Tier I meeting

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Ongoing Action Items					
Action Item No.	Responsible Party	Status	Due Date	Action Item	Comments
A-0209-02	Y. Martínez	Completed		Take first step in developing exit strategy to be ready for May Tier I & June Tier II meeting.	The draft has been completed for discussion at the next Tier I meeting.
A-0209-04	B. Fisher	Completed		Will contact Helen concerning contracting CH2MHill for Tier I participation.	CH2M Hill will be added to the Tier I. Funding issues still to be resolved.
A-0209-05	G. Crane	Completed		Gather fact sheets and other info for Sun Herald via PAO.	
A-0209-06	G. Crane	Completed	2/16/09	Follow up on Fred Boykin concern through a phone call	Gordon contacted Mr. Boykin and his concern was not related to NCBC. Referred to USACE.
A-0209-07	G. Crane	Completed		Request additional copies of PHA.	Requested but not yet received. Gordon will make copies if needed.
A-0209-08	G. Crane & N. Rouse	Completed		Confirm new RAB mtg. location.	
A-0209-12	B. Fisher	Ongoing	2/20/09	Talk to Steve Beverly concerning notifying land owners about the completion reports.	Steve requested to review documents prior to sharing the document with land owners.
A-0209-13	B. Fisher	Ongoing	2/12/09	LTM Planning and funding Site 8 sediment.	
A-0209-14	N. Rouse	Ongoing	2/13/09	Confirm community relations actions needed for site 8c.	
A-0209-15	B. Fisher	Completed		Schedule discussion/meeting with optimization team for site 10 to complete report.	The meeting has been held. The remedy will include placing a culvert in the ditch and revising existing reports. However, funding is not available at this time to revise the documents.

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Ongoing Action Items					
Action Item No.	Responsible Party	Status	Due Date	Action Item	Comments
A-0209-16	B. Fisher	Completed		Will review pilot scale sites 3 & 4 funding.	Optimization team determined that the concentrations at Site 3 were too low for enhanced bioremediation. A pilot study will be funded for Site 4.
A-0209-17	B. Fisher	Complete	5/12/09	Resolve Edwards property site restoration issues (letter from the state).	Overcome by events. MDEQ has sent a letter concurring with the investigation results.
A-0209-19	N. Rouse	Ongoing		Find BOP dates and forward to team.	Nancy will forward email regarding updated BOP dates.
A-0209-20	N. Rouse	Ongoing	4/17/09	Nancy will find the EPA training link and forward the link to the team	
A-0312-01	G. Crane	Completed		Get information about the West Side Community Center	
A-0312-02	N. Rouse	Completed		Provide information to Gordon in order to assist him to get the contact for the Public Assessment Study	
A-0416-01	G. Crane	Completed	4/27/09	Secure RAB meeting room	
A-0416-02	B. Fisher	Completed	TBD	Identify future funding for CH2M Hill Tier I participation	
A-0416-03	Y. Martinez	Completed	4/27/09	Provide surface soil data from Site 6 to Gordon Crane and Bob Fisher	

Note:

Shaded rows have been noted as “Completed” and will be removed from the Ongoing Action Items Table prior to the next action item review.

*Action Item A-0509-01: Robert Fisher - Send Bob Merrill the updated portions of the completion report for Canal Rd. (Due 6/4)*

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*Action Item A-0509-02: Robert Fisher - Verify the correct process (is there something needed for the landowner to close the investigations) to close the investigation (final step) for Canal Rd with Steve Beverly. (Due 6/4)*

During review of Ongoing Action Item A-0209-17 Gordon noted that the Navy never had a signed access agreement with Edwards; there had been language in a drafted agreement concerning keeping the road in usable condition, as well as language addressing the pond, however, nothing was ever put together and officially signed.

The next RAB would be in August 10, 2009. The Navy will coordinate appropriate funding for the RABs to continue without any interruptions.

*Action Item A0-509-03: Gordon Crane - Verify RAB administration funds (for meeting place, signs, etc.) (Due 6/4)*

## **5. Lunch**

## **6. Contractor Issues – Bob Fisher**

The Navy provided a brief contract update. Due to funding issues CLEAN IV modifications and task orders have been put on hold. However, there is a new contract vehicle for TtNUS, the CLEAN IV contract, which is actually referred to as CLEAN 1001.

TtNUS expressed concern over Site 6, on which there is quarterly monitoring; it was supposed to have a second year of monitoring.

Helen clarified that the CLEAN contracts basically cover site studies, while RAC contracts are passed along to the contractors to begin the work. CLEAN and RAC are both cost plus contracts. Gordon asked whether or not the Navy was keeping the EMAC program.

Helen replied that the Navy was keeping the EMAC program, but that in the mean time they are going to have an alternate environmental contract as a temporary fix.

Bob Fisher and Helen briefly described the CLEAN 1001 contract, noting that TtNUS had the contract, and that it was being managed out of NAVFAC Atlantic. They intend to use the same project managers, but additional administrative details will be discussed later.

## **7. Round Table Discussion (Joint Tier I/Tier II Meetings, Outstanding Action Items/Loose Ends)**

Mrs. Lockard explained that the Tier II intent in proposing a joint Tier I/II meeting was to benefit the Tier I team. The joint Tier I/II meetings topics have included team success stories, technical presentations, and training. The joint meetings have also presented an opportunity for networking and discussing new technologies.

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Bob Merrill posed the question of whether or not this type of meeting would really be beneficial to the team. Mr. Merrill expressed his concern that an additional meeting would put him further behind on document review.

The team discussed the topic further, noting that the meeting would need to be tailored to be relevant to the Gulfport Partnering Team. Nancy suggested taking a moment to brainstorm to come up with potential joint Tier I/II meeting topics:

- What is the future of MDEQ's relationship with the Navy? (MRP Sites?)
- Lessons Learned (other partnering experiences)
- Clarify Tier I/II standard communications (MS)
- Identify how other Tier I/II teams communicate
- Clarify what does the Tier II team expects Tier I to bring to the table
- Can lines of communications be opened between Tier I & II members?
- Success stories from each team
- Networking
- Technology topics (ex. sustainable remediation, multi-increment sampling, listing of sites: challenges and types of RA, site closeouts, LUCs)

## **8. Break**

Nancy noted that the last discussion was heavily team building. Because of this, the time allotted to the partnering exercise agenda item was reduced. Nancy provided a brief review of the agenda since discussion was running behind schedule.

*Action Item A-0509-04: Bob Fisher - Verify legal requirements related to RCRA Permit on Site 8 with Steve Beverly. (Due 8/11)*

## **9. Exit Strategy/Long- and Short-term Goals – Robert Fisher**

Mr. Fisher explained that for years Gulfport has not had an Exit Strategy. Instead the Team kept a list of priorities, which has worked well at the Tier I level, but hasn't been a great way to convey information to Tier II. The Exit Strategy is really a tool to communicate when we need to bring activities to a close and how to measure our success. These things weren't expressed to us by our last Navy RPM; we just understood generally when he received pressure from his superiors.

Mr. Fisher and Yarissa provided additional information on the questions answered/details provided in the Exit Strategy:

- What's in progress?
- What is the contractor currently working on?
- What phase is the site in?

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Bob Fisher pointed out that the Remedy In Place (RIP) dates are the dates projected in the NORM database. The RIP date doesn't have to be the final remedy, it can/should be the initial action.

The Team began a site by site Exit Strategy review:

#### Site 1: Disaster Recovery Area

The RI is in progress (TtNUS); it has not yet been submitted.

Mr. Fisher and Yarissa agreed the forecasted RIP date was not realistic. Helen stated that if the site ranking was at medium, then the RIP date could be pushed back to 2011.

Gordon noted the site has a lot of problems concerning drainage, infrastructure needs, etc. Bob Fisher replied that he had walked the site with the drainage engineer. Site 1 is basically a bowl; the soil fill required to change this is cost prohibitive.

*Action Item A-0509-05: Gordon Crane – Verify NCBC plans/needs for Site 1 (footprints). (Due 5/26)*

Once the exit strategy had been finalized and reviewed at the Tier II meeting, the Navy will update the NORM database.

Mr. Fisher stated that the plan included cover, LUCs, and monitoring. Concerns/barriers: long term site plans at NCBC and funding issues.

The Team took a moment to review the site rankings:

High – Sites 6 and 10

Medium – Sites 1, 2, 3, 4, 5, and 8

Low – Site 7

Helen stated that once the Baseline date is established, it does not change.

#### Site 2: World War II Landfill

TtNUS has just started the RI.

Bob Fisher stated that assuming no plumes were found, and assuming the SAP goes through the first time, TtNUS could begin to write the RI Work Plan in the next month.

Concerns/barriers: Trying to accommodate the golf season, irrigation issues, wetland issues (Magnolia Bay).

#### Site 3: Northwest Landfill/Burning Pit

The Site 3 RI is a priority.

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Concerns/barriers: golf course, new housing construction  
Site 4: Golf Course Landfill

Bob stated that work has been started on the RD/FS/RI – these had been stacked up.  
Helen noted that this should probably be considered the highest priority. The remedial action is in place because the funding was available.  
Mr. Fisher stated that the PP could be written before the RA process finished.

Concerns/barriers: Need to work close with the gulf club house, which will impact their business. There are also two bridges that will be a concern. Drainage should not be an issue; permitting is in place to line it. Gordon noted that an irrigation system would be put in, stopping at the edge of Site 4.

Site 5: Heavy Equipment Training Area Landfill

Remedial Action is in progress.  
Concerns/barriers: Establishing the vegetative cover (getting the grass to grow).

Site 6: Fire-Fighting Training Area

No Decision Document (DD) for Site 6; the action memorandum in July of 2008 incorporated the two system changes and updated that.  
Comments: The action memo was revised in 2008 (use the original memorandum date for the DD baseline)  
Concerns/barriers: Parking area being built on the site; 2 of 5 monitoring wells will be affected.

*Action Item A-0509-06: Gordon Crane – Verify the dates of the installation/removal of the response system at Site 6. (Due 5/26)*

Site 7: Rubble Disposal Area

ESI in progress (did an ESI in 1999).  
Bob Fisher stated that there were breakdown products from solvents. It may be naturally attenuating.  
Concerns/barriers: VOCs and the golf course

Bob Merrill asked what happens if these deadlines are not met.  
The Navy explained that they could lose their funding. Mrs. Lockard explained that they will be asked why things weren't finished when the funds were available; NCBC Gulfport in particular has so few sites compared to other bases that it would be difficult to justify not meeting the deadlines.

Site 8: Air Force Herbicide Orange Storage Area

Site 8A: The DD is in progress but has not been signed.

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Site 8B&C: The DD is in progress.

Bob Fisher stated that his plan was to use the LTM to tie up 8b&c and get the DD in place. Mr. Fisher explained that the intent of the LTM of the sediments was to have an early warning sign of dioxins moving downstream.

Further Team discussion concerning a LUC RD led to the agreement that Sites 8a, 8b, and 8c would be covered by a single LUC RD document.

#### Site 10: PCB in the Ditch

Removal action was performed in April of 2000 (need to check the date); this counts as the RIP date.

Yarissa asked whether or not a DD was necessary. Bob Fisher replied that it had been a removal action, not a remedial action.

Concerns/barriers: utilities (gas); medical clinic nearby

*Action Item A-0509-07: Gordon Crane & Nancy Rouse – Provide Tetra Tech NUS, Inc. a copy of the administrative record. (Due 5/13)*

*Action Item A-0509-08: Yarissa Martínez – Complete the exit strategy and email the team for consensus.*

## **10. Closing**

Nancy asked the Team whether or not they wanted to do the Partnering Exercises in the morning as part of or in lieu of the check in. The team had no objections to having the Partnering Exercises in the morning. Nancy asked the team to prepare an answer to the question “What was the most difficult or important challenge of your childhood?”

### **1. 2nd Day Check In**

Each meeting attendee provided a brief personal update.

### **2. Partnering Exercise**

**Where did you grow up?**

**How many kids were in your family?**

**What was the most difficult or important challenge of your childhood?**

### **3. Site 2 DQO’s – Peggy Churchill (TtNUS)**

Peggy Churchill explained that she was providing an introduction to the UFP-SAP since this would be the Team’s first exposure to it. Mrs. Churchill stated that she would be providing an overview, discussing the origins, introducing the 7 Steps, and reviewing the Conceptual Site

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Model (CSM). Depending on site complexity and the functionality of the group, the DQO process can take several days. It is expected that the Team should be able to get through Step 4 before lunch.

Mrs. Churchill briefly explained the TtNUS review process, noting what happens before it gets to the Navy chemist. Each attendee provided feedback concerning their level of experience.

Mrs. Churchill explained that the idea behind the process was to protect the Team from having issues down the road. The idea is to get input from everyone, so that you know what to do with the data collected. The point is to capture goals, objectives, issues, and resources; it is purpose driven; it clearly defines the problem through the agreed upon CSM, it identifies goals, and it focuses on the desired end state and exit strategy. These things are important to identify, in order to know when things can be considered complete/finished.

Peggy provided background information on the origin of the UFP-SAP with a Power Point Presentation. The UFP-SAP was developed by the IDQTF Workgroup in response to an IG report on data quality. This work group included the EPA, DOD, and DOE.

#### UFP-SAP Elements

- Problem solving and objectives
- Conceptual site model
- Sampling design and rationale
- Action levels and analytical methods
- Verifications, validation, and usability
- Exit strategy

#### DQO Overview

- Problem Statement - What questions are you trying to answer?
- Study Goals
- Information Inputs - all info used in this investigation
- Study Area Boundaries - sometimes this is predefined
- Decision Rules - how we use the data
- Performance and Acceptance Criteria - statistics used to determine how many samples are needed to be representative.
- Data Collection Plan - sampling plan

Mrs. Churchill projected a figure of Site 2 and asked Mr. Fisher for explanation of the site since the team members had not been there. Mr. Fisher provided Site 2 background information stating that the site had been a trench and fill landfill; they used to burn at this site (accelerant was used). Previous documents state that daily burns took place, but others said that was not the case. There many different types of things disposed there. The groundwater is around 4-6 ft bls. When the IAS was performed in 1985 the assumption was that the groundwater flowed to the south, and installed wells accordingly. The groundwater actually flows north-northwest and changes seasonally. A monitoring report was completed in 1997. In 1998 monitoring wells were installed downgradient.

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Mr. Fisher went on to say that this is a presumptive remedy, so it does change our approach and how much data we'll need. What we have at this site is very little flow; another issue is the significant layering of silt/sands at the surface. It should also be noted that across the street at Site 3 (to the west) there was some chlorinated solvent that created some very bizarre plume maps. Due to that experience, these are some things we'll be looking for at Site 2.

Mr. Fisher stated that the 1985 data was useless since it was collected upgradient. Groundwater samples were collected in 1998 from the downgradient monitoring wells. Afterwards a CSM was developed. The thought was that an RI would be completed quickly, but it didn't happen.

Peggy asked when the golf course was built. Bob Fisher replied that it happened in 1997 or 1998.

Bob noted that because of the fill placed at the site (0-8ft), a pond was created; there had been a wet area, which was turned into a course feature. This changed the hydrology somewhat.

Mrs. Churchill asked what happened to the soil that was moved in order to create the pond. Gordon Crane responded that it had been put into borrow pits which were used onsite.

TtNUS asked if the 1987 Confirmation Study data could be used. Mr. Fisher replied that it could be incorporated, but that it wouldn't really change the number of samples. The 1987 monitoring wells were destroyed during the golf course construction. The problem with the geophysical study was the wide grid. There were big sediment studies in the 1990s which covered this area, and a delineation report. A lot of data was collected, but it was really focused on the Herbicide-Orange.

Gordon asked whether or not the wetland might act as a reservoir for anything. Mr. Fisher replied that sampling would take place there.

Bob Fisher asked whether or not the bunkers were still being used. Gordon replied that they were still in use.

Mr. Fisher noted that access can be an issue. There are a lot of unknowns.

*Action Item A-0509-09: Gordon Crane – Provide the team with aerial pictures related to Site 2.*

Mr. Fisher explained that the presumptive remedy in this case will be a cover over the landfill. There is guidance as to what the cover needs to be. This would be the fourth of the landfills that have been covered at Gulfport.

Mr. Fisher noted that this is a reducing environment.

The Team reviewed the CSM.

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*

### Sources:

- Construction debris (preserved wood – DPT issues)
- Solvents
- Paints
- Coal-tar Distillate
- Dioxins – from burning
- Might have some medical waste out there
- General municipal wastes

Gordon brought up information concerning a possible MRP site. A pistol range was located to the north of Site 3. The berm may have been on Site 2.

*Action Item A-0509-10: Gordon Crane – Send the team information concerning the potential MRP site.*

Yarissa asked Gordon what was stored in the bunkers.

Gordon replied that they used to store small ordnance; two of the bunkers are still actively used.

*Action Item A-0509-11: Gordon Crane – Send Yarissa information regarding the bunkers (former small ordnance storage area) at Site 2.*

Yarissa expressed concern regarding the possibility of small ordnance at the bunker locations in relation to field personnel safety.

### Primary Media

- Surface soil
- Bottom of waste soils in contact with groundwater

### Migration Pathways:

- Seasonal fluctuation of groundwater
- Infiltration
- Runoff/erosion
- Surficial aquifer vs. deeper aquifer (20-30 ft bls)

### Secondary Media

- Surface water, groundwater, surface & subsurface soil

Peggy asked whether or not MDEQ makes a determination on drinking water status. Bob Merrill replied that they did not.

### Receptors

- Ecological
- Human (screen residential – Tier 1 TRGs which are essentially the same as the EPA residential; recreational use, trespassers, industrial/construction workers, people fishing).

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*

Mr. Fisher replied that risk at other landfills in the base has been very low. The only issue was concerning the consumption of groundwater.

The Team discussed the complete/potentially complete secondary sources/media. If feasible, the cap will be as close to a RCRA model cap as possible.

Exposure Pathways (the way the receptor will be exposed or potentially at risk)

- Ingestion of groundwater
- Dermal Exposure
- Inhalation

**STEP 1: PROBLEM STATEMENT**

What environmental question are we trying to answer?

What type of investigation is it?

What media will be investigated?

Are there any COPCs? Can we narrow down the analytes list?

Questions:

Are humans/ecological receptors at risk from exposure to contaminated media (groundwater, surface water, surface soil, subsurface soil, sediments) at the site?

Does the site meet presumptive remedy requirements?

Will the presumptive remedy (landfill cover) remedy/mitigate risk?

Presumptive Remedy Municipal Landfill – Team Review

Determine whether or not the containment presumption is appropriate.

- What do you do if the answer is no? – specialized conditions noted
- If yes? – go with presumptive remedy

Highlight 1 – containment strategy

- landfill cap
- source area groundwater control to contain plume
- leachate collection and treatment
- landfill gas collection and treatment, and/or
- institutional controls to supplement engineering controls

RAOs

Presumptive remedy RAO:

- prevent direct contact with landfill contents
- minimizing filtration and resulting contaminant leaching to groundwater
- controlling surface water runoff and erosion
- collecting and treating contaminated groundwater and leachate to contain the contaminant plume and prevent further migration from source area; and
- controlling and treating landfill gas

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*

Non-presumptive:

- remediating groundwater
- remediating contaminated surface water and sediments; and
- remediating contaminated wetland areas

Peggy explained that the overall study goal is to determine if containment is appropriate. If it isn't then you almost need to have another assessment/investigation.

Mr. Fisher stated that when the USEPA looked at the RODs you could see that the caps dominated the technologies applied.

## **STEP 2: STUDY GOAL**

What are the main objectives of this investigation?  
How will the environmental questions get answered?

The study goal is written as an "if, then" statement.

Determine whether or not the containment assumption is appropriate (flow chart).  
The secondary study goal is risk assessment.

## **STEP 3: INFORMATION INPUTS**

What type of data and information is needed in order to achieve the study goals?  
What information should be collected?  
How may land reuse plans affect remedy selection?  
Determine old data that is usable, what new data will you collect?

The Team noted that due to the nature of the sources the analyte list for Site 2 will be lengthy.  
Bob Merrill suggested starting with a target compound list.

### Chemical Data:

TCL VOCs & SVOCs

PAHs

TRPH (will address with the state)

TAL metals;

Dioxins? – Bob Fisher stated that it may not be necessary to test all the media for landfills; selective analysis for dioxins would probably be a good idea, but could be limited to surface soil, groundwater and sediment in select locations

Pesticide/herbicides

PCBs – limited investigation

Total Organic Carbon – surface soil

Mr. Fisher noted that the team should define the site dynamics, and highly immobile contaminants.

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*

Mrs. Churchill asked whether or not the presumptive remedy addressed monitoring issues. Mr. Fisher replied that the presumptive remedy doesn't really include details concerning long term monitoring. Previously, we applied the Michigan model for statistical analysis to the total number of sampling points, and then broke it down into subdivided areas. The end result was about 15-20%, so we're going to have a lot of volatiles. Then we selected points for the less mobile contaminants.

#### Physical Data:

Six field parameters for groundwater.

- 1a. Geotechnical parameters: limited to hydraulic conductivity and grain size (unless we were doing load bearing issues, which isn't anticipated) Geophysical study will help determine boundaries of landfill.
- 1b. Soil/Gas Study – what cells have hot spots?
2. DPT – surface soil, subsurface soil, groundwater – from stinger and temporary wells in select locations; PID screening – subsurface samples
3. Permanent wells – sediment, surface, wetland area  
Slug/Aquifer Testing

Peggy asked how the geophysical survey would impact sample location. Mr. Fisher replied that the geophysical data is really good for providing physical boundaries, but it doesn't tell you which cells have mobile/immobile contaminants. A soil gas study helps refine the geophysical study info.

Yarissa noted that there had been some TtNUS concern regarding the possibility of geophysical/soil-gas study issues/equipment interference.

Bob Merrill informed the Team that the Mississippi Geological Survey will e-log soil borings for free, if coordination takes place prior field event commencement.

Peggy asked how many wells would be advanced to the deep aquifer. Mr. Fisher replied that there would probably be no more than four. Peggy noted that this would help define the vertical boundary.

#### **STEP 4: STUDY AREA BOUNDARIES**

Horizontal Boundary: The boundary will be determined by the results of the geophysical study, unless a Phase II investigation is required (the current boundary is based on areal photos and geophysical results, indicating the waste disposal area).

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*

Vertical Boundary: Soil – surface soil 0-1 ft (potentially 2 intervals); subsurface soil 1-6 ft (or to water table); groundwater – mostly surficial, a few intermediate or deep (up to 60 ft); sediment – 0-6 inches.

The team had a brief discussion concerning sediment sampling depths. Mr. Fisher explained that vertical profiling of the ditches took place.

Mrs. Churchill noted that SPLP had not been discussed earlier. Mr. Fisher replied that typically they haven't been doing SPLP; if they were considering not putting on a lower permeability cover, then they might agree that SPLP should be done.

#### **STEP 5: ANALYTIC APPROACH**

Step 5 will be based on the presumptive remedy guidance and the Michigan regulations (see previous work at site 3).

Peggy stated that individual sampling results would be compared to the criteria.

Groundwater – individual sampling results will be compared the screening data  
Soil – 95% UCL Tier I TRGs

Helen asked if MDEQ had a Tiered approach. Bob Merrill replied that they did for screening, but that they enforced the MDEQ standards.

Bob Fisher suggested a review of how things were addressed at Site 3.

Peggy stated that the Team still needed to come up with a sampling approach that provides a representative sample and a properly delineates the site.

Mr. Fisher explained that you delineate, generally when its near a boundary and/or appears that it may migrate; the way it worked out it was more than we originally thought, at Site 1 it was closer to 30. At Site 3 we may be in the 80-90 range because of some hotspots.

Mr. Fisher stated that a sampling plan would be proposed informally before it came across in a document.

#### **4. Meeting Closeout**

Action Item Review: See Attachment

Nancy informed the Team that the Basics of Partnering (BOP) Training would be June 11-12<sup>th</sup> in Tampa. Nancy will forward the information to the Team via email.

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## MEETING REVIEW

<u>+ (pluses)</u>	<u>Δ (deltas)</u>
The room	Uncomfortable chairs
Good roundtable discussion	Off-schedule
Dinner/Lunch with team	Confusion concerning agenda
DQO discussion was interesting	Meeting room change
Level of preparation to speak about topics	
Good participation	
Accomplished goals	

### Confirm meeting/conference call dates:

RAB Meeting: August 10<sup>th</sup> (PP for Site 4 public meeting)

Partnering Meeting: August 11-12<sup>th</sup>

Teleconference: June 9<sup>th</sup> & July 7<sup>th</sup> – 1pm CST

Nancy asked the Team whether or not they'd prefer to use the same meeting space.

TtNUS will need to review the budget and check for availability. Mr. Fisher will assist TtNUS.

### Facilitator Feedback – Nancy Rouse:

Nancy provided facilitator feedback, noting the positive and open communication. Nancy noted a need to review/modify the ground rules and Team Charter.

*These minutes are a summary based on informal notes taken at the meeting. They are not intended as a verbatim transcript and may not have captured everything that was discussed.*



**TETRA TECH**

## Introduction to the UFP-SAP and Data Quality Objectives/Project Planning

World War II Landfill- Site 2  
Remedial Investigation  
Data Quality Objectives/Project Planning

**NCBC Gulfport, MS  
Partnering Meeting, May 13, 2009**



## What is the UFP-SAP?

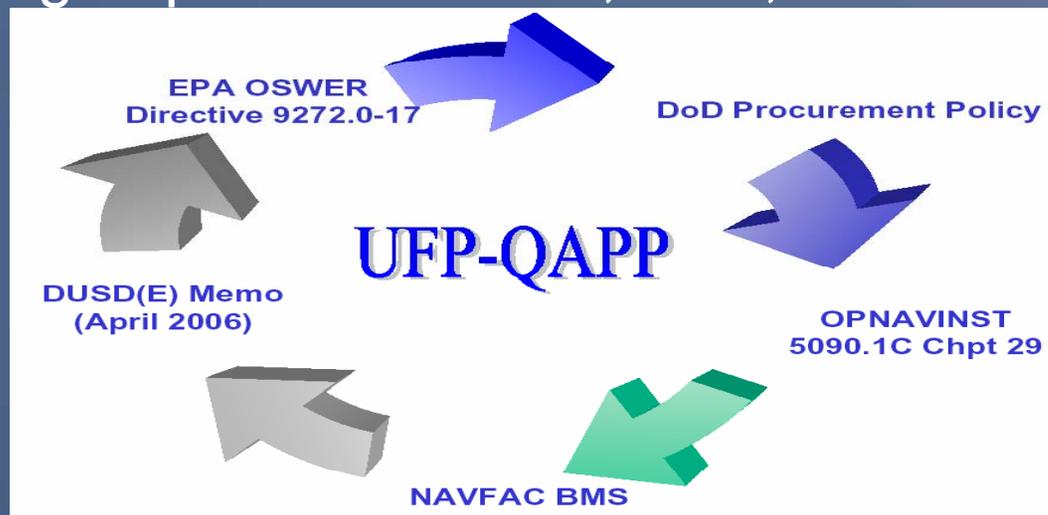


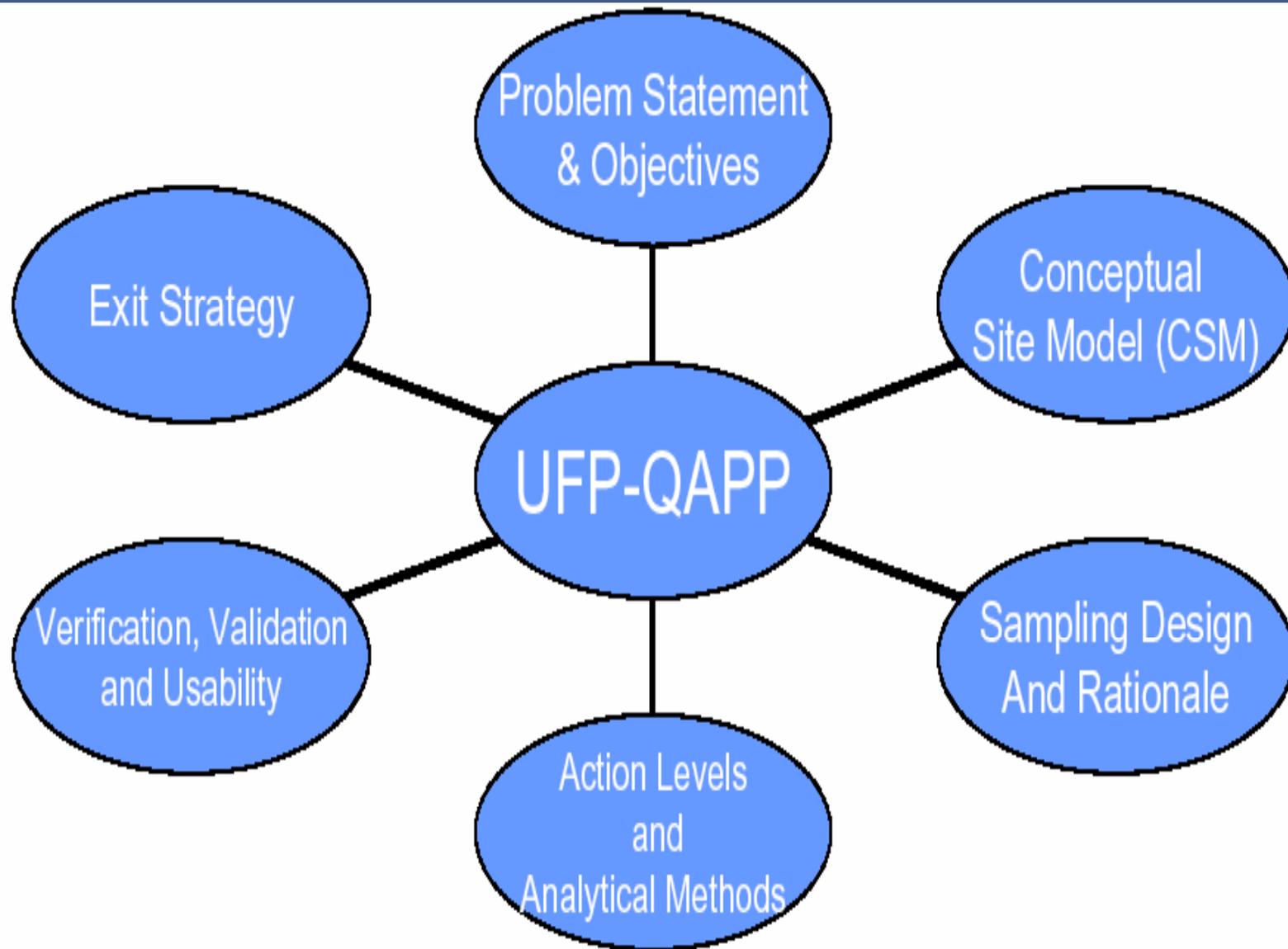
## How Can it Help Our Team?

- Team based approach to project planning, incorporates input from all stakeholders
- Project management tool
- Captures and documents: Project Goals, Objectives, Issues, Schedules, Resources
- Purpose Driven:
  - Clearly defines the problem through agreed upon Conceptual Site Model
  - Identifies goals
  - Focuses on desired end state
  - **EXIT STRATEGY!**

## Origin of the UFP-SAP

- Documents and integrates all technical and quality aspects of a project throughout its lifecycle
- Developed by the Intergovernmental Data Quality Task Force (IDQTF) Workgroup in response to an IG report on data quality
- Workgroup included EPA, DoD, and DOE





## DQO Overview

- Problem Statement
- Study Goals
- Information Inputs
- Study Area Boundaries
- Decision Rules
- Performance and Acceptance Criteria
- Data Collection Plan



DRAWN BY K. MOORE	DATE 5/8/09
CHECKED BY Y. MARTINEZ	DATE 5/8/09
COST SCHEDULE AREA	
SCALE AS NOTED	



LOCATION MAP  
SITE 2 - WORLD WAR II LANDFILL  
NCBC GULFPORT  
GULFPORT, MISSISSIPPI

CONTRACT NUMBER CTO 0150	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. FIGURE --	REV 0

## Conceptual Site Model

- Sources
- Primary Contaminated Media
- Migration Pathways
- Secondary Contaminated Media
- Receptors
- Exposure Pathways

# Site 2 CSM

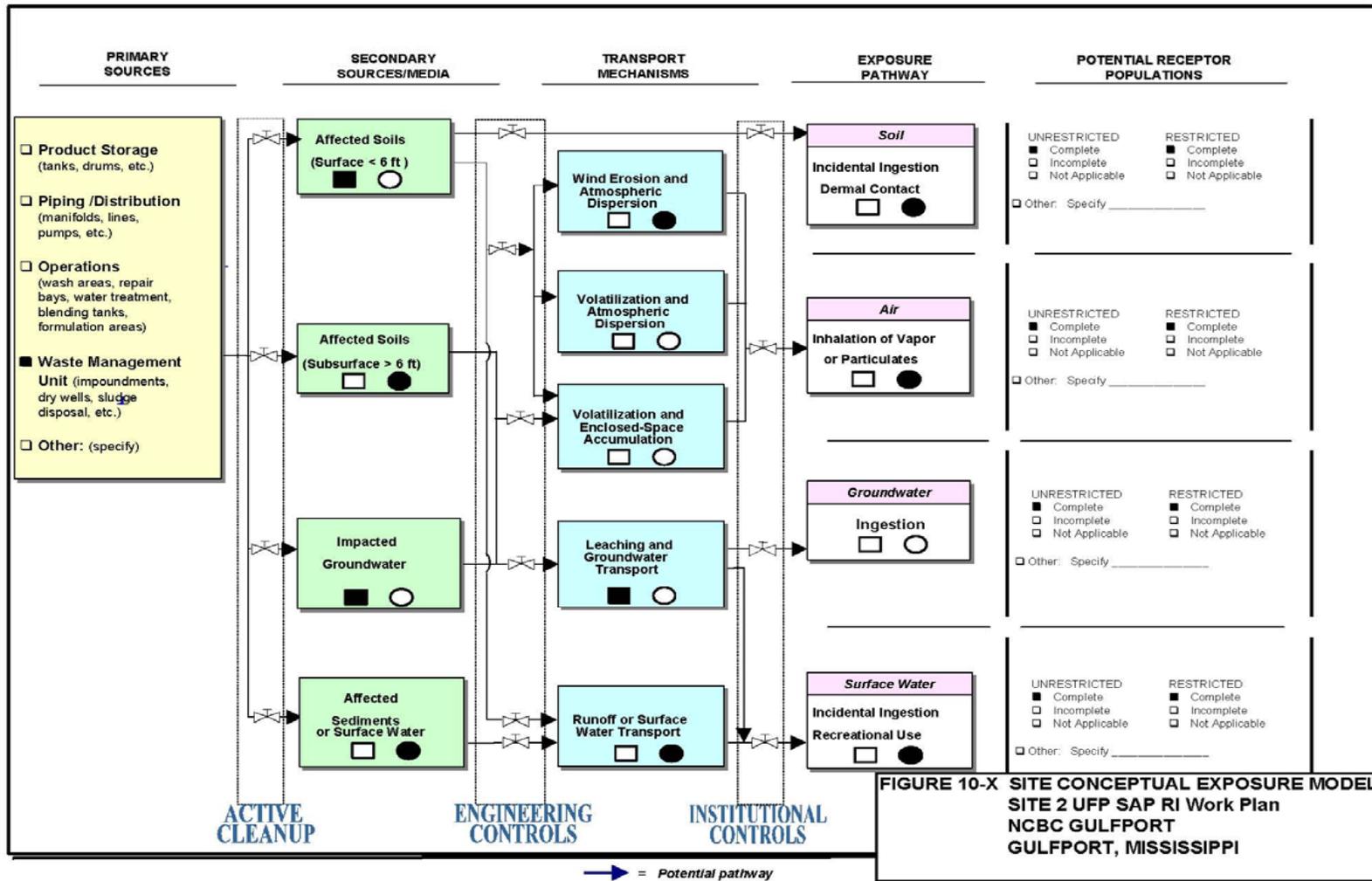
## Worksheet

## Baseline Site Conceptual Exposure Model (SCEM)

Site Name: **Site 2, World War II Landfill**  
 Site Location: **NCBC Gulfport, Mississippi**

Completed By: **YML**  
 Revision Date: **5/04/2009**

Complete  Draft  
 Potentially Complete  Final



## Step One: Problem Statement

- What environmental question are we trying to answer?
- What type of investigation is it?
- What media will be investigated?
- Are there any COPCs? Can we narrow down the analyte list?

## Step Two: Study Goals

- What are the main objectives of this investigation? How will the environmental question get answered?
- Written as an if... then.... statement. What occurrence will trigger an action?

## Step 3: Information Inputs

What type of data and information is needed in order to achieve the study goals and make decisions at the sites?

- Any previously collected data
- Geophysical Survey
- Chemical Data
- Physical Data
- Project Action Limits

## Step 4: Study Area Boundaries

- Describe Study Area
- Describe Horizontal Boundary
- Describe Vertical Boundary
  - Soil Intervals of Interest
  - Depth to Water Table

## Step 5: Decision Rules

- Exactly how will the study goals be achieved- refine step 2.
- Are individual sample results going to be compared to screening values?
- Averages or 95% UCL?
- How to decide if a constituent is a COC?

## Step 6: Performance and Acceptance Criteria

- What type and how much error is acceptable?
- Statistical analysis
- Biased vs. random sampling
- Representative sample number

## Step 6: Performance and Acceptance Criteria

- Professional judgment
- Knowledge of the Conceptual Site Model (CSM)
- Visual Sampling Plan (VSP)

## Step 7: Data Collection Plan

- Sample Design and Rationale
- Describe all types of sampling and all media being sampled

## **APPENDIX B**

### **HISTORICAL BACKGROUND INFORMATION**



39501 - IRP  
01.01.02.0001

December 22, 1994

Commanding Officer  
Southern Division  
Naval Facilities Engineering Command  
2155 Eagle Drive  
North Charleston, SC 29418

Attention: Mr. Art Conrad

Subject: Observations at Site 2 during basewide groundwater sampling

Dear Art:

Bob Fisher has just returned from the basewide sampling event and has related to me some observations he made at Site 2 during his time at NCBC. I thought that we should relay these to you ASAP.

There appears to be true rainbow sheen in the ditches to the south and to the east of Site 2. Bob followed the east ditch to the north into the wooded area and noticed severely distressed vegetation (dead trees) along with sheen. The sheen in the south ditch was noted only on the north (site) side of the ditch. Sheen in the ditch along the eastern edge of the site was noted on both sides of the ditch. The dead trees included both pine and deciduous.

The south side of the site is along gradient and the east side is upgradient. It is not surprising to find the southern sheen, but the presence of sheen in the east ditch is somewhat unsettling.

If you have any questions, please call me at (615) 531-1922.

Sincerely,

ABB ENVIRONMENTAL SERVICES, INC.

Penny M. Baxter  
Senior Project Manager

attachment

cc: file

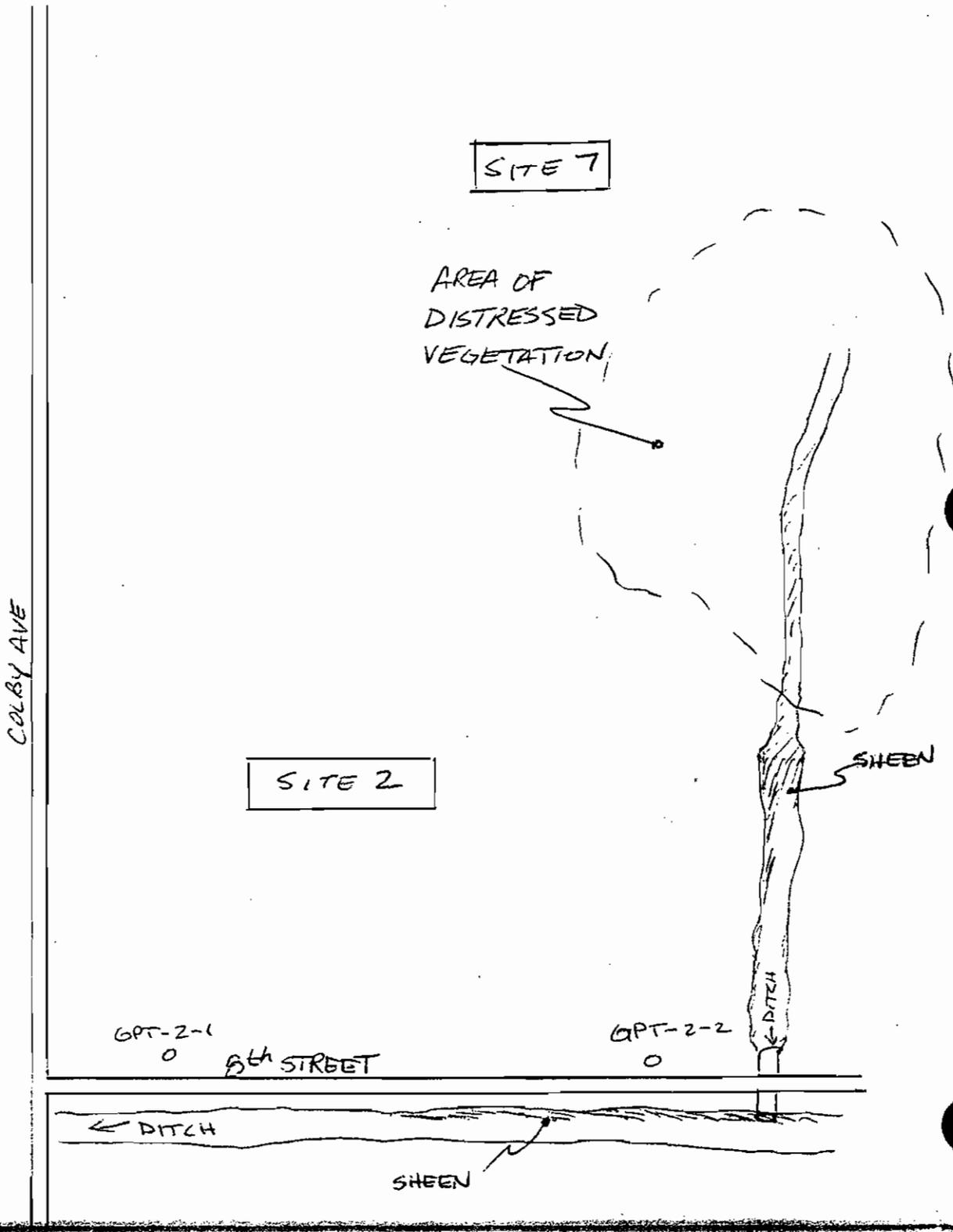
[8505.0<sup>50</sup>~~48~~]

ABB Environmental Services Inc.

PROJECT  
NBCG GULFPORT  
FIELD OBSERVATIONS

COMP. BY  
R.F.  
CHK. BY

JOB NO.  
DATE  
12-22-94



39501-GENERAL

01.02.00.0003

A Report Prepared For

Department of the Navy  
Southern Division  
Naval Facilities Engineering Command  
2155 Eagle Drive  
North Charleston, South Carolina

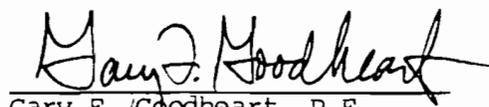
FINAL VERIFICATION REPORT  
NAVAL CONSTRUCTION BATTALION CENTER  
GULFPORT, MISSISSIPPI

HLA Job No. 2176,093.12

ADMINISTRATIVE  
RECORD

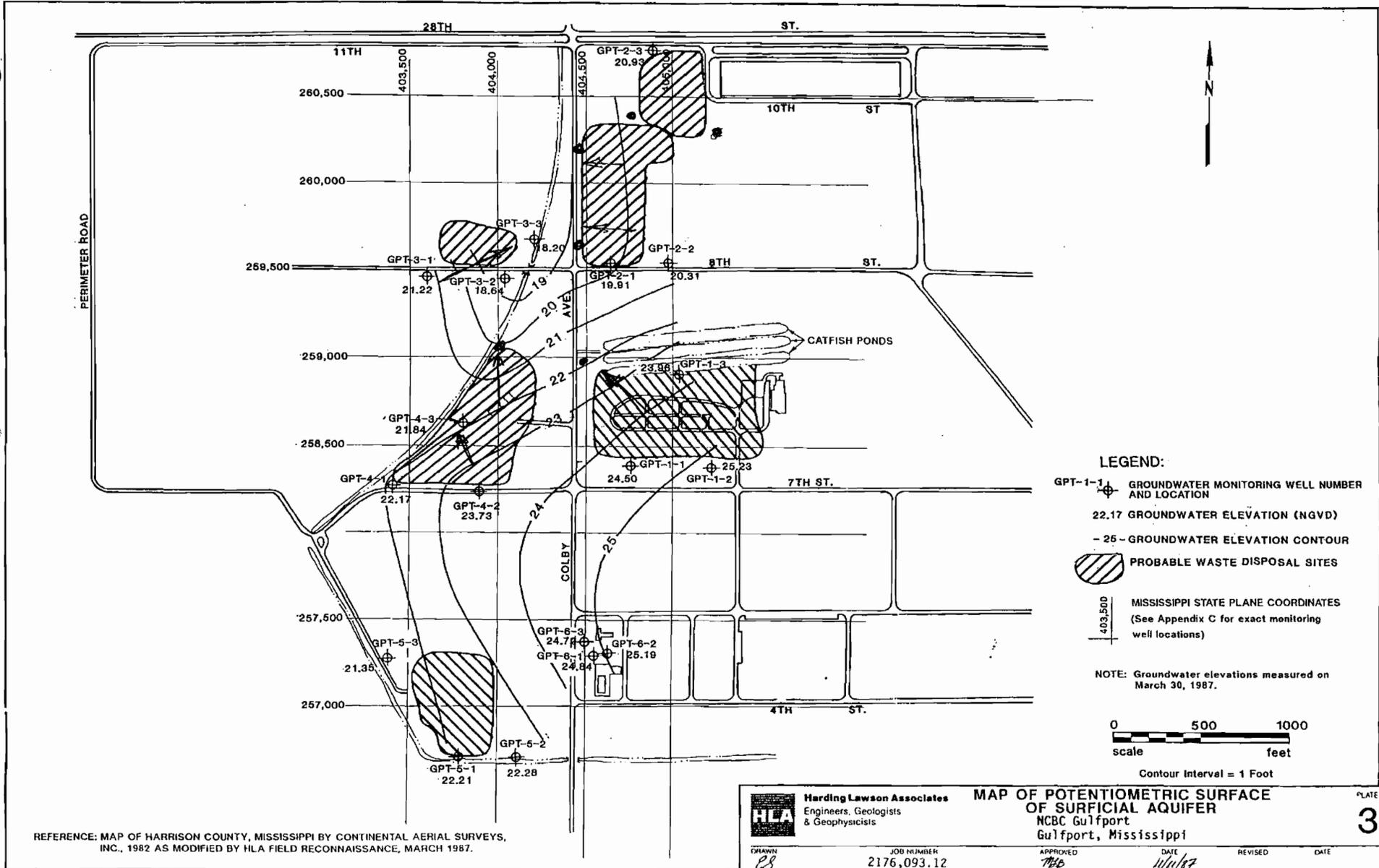
by

  
Michael L. Bergstrom  
Senior Hydrogeologist

  
Gary F. Goodheart, P.E.  
Principal Engineer

Harding Lawson Associates  
6220 Westpark Drive, Suite 100  
Houston, Texas 77057  
Telephone: (713) 789-8050

July 7, 1988



REFERENCE: MAP OF HARRISON COUNTY, MISSISSIPPI BY CONTINENTAL AERIAL SURVEYS, INC., 1982 AS MODIFIED BY HLA FIELD RECONNAISSANCE, MARCH 1987.

PLATE

3

TABLE 8  
SUMMARY OF CHEMICAL ANALYSIS RESULTS, SITE 2

Location	GPT-2-1	GPT-2-2	GPT-2-3	SW2-1	SD2-1	Decon 1	Decon 2
Sampling Date	3/28/87	3/28/87	3/28/87	3/26/87	3/26/87	4/7/87	4/7/87
Temperature	20	18	17	20	-	22	21
pH (field)	5.99	5.49	5.19	7.41	-	7.42	6.78
Specific Conductance (field)	230	210	660	270	-	245	20
pH (laboratory)	6.42	5.66	5.61	7.52 (7.48)	5.80	8.83	5.85 (5.89)
Specific Conductance (laboratory)	160	120	440	200 (200)	-	490	6
TOC	-	-	-	10	2,800	3	< 1
TOX	-	-	-	73	300	7	11
COD	-	-	-	40 (35)	8,240	24	19
O and G	-	-	-	1.5	< 123	1.5	1.7
Cd	< 4.7	< 4.7	< 4.7	< 4.7 (<4.7)	<2.9 (<2.9)	< 4.7	< 4.7
Cr	26	73	21	< 7.8 (<7.8)	4.9 (<4.8)	< 7.8	< 7.8
Pb	20	41	13	< 5.0 (<5.0)	4.7 (3.7)	< 5.0	< 5.0
Volatile Organics	(1)	(1)	(1)	-	-	(1)	(1)
1,2-trans-Dichloroethylene			37				
Trichloroethylene			5				
Toluene			1*				8
Chloroform						14	
Dichlorobromomethane						2*	
Acid/Base/Neutrals	(1)	(1)	(1)	-	-	(1)	(1)
bis (2-Ethylhexyl) Phthalate			21				

Note: 1. All analyses results for water samples are reported in  $\mu\text{g/l}$  except TOC, COD, and O and G which are reported in  $\text{mg/l}$ . Analyses results for sediment and soil samples are reported in  $\text{mg/kg}$ . Temperature, pH, and specific conductance are reported in  $^{\circ}\text{C}$ , units, and  $\mu\text{mhos/cm}$  at  $25^{\circ}\text{C}$ , respectively.

2. Results presented in parentheses are for duplicate analyses.

3. Temperature, pH (field), and Specific Conductance (field) data for groundwater samples are an average of three separate measurements.

(1) All chemical parameters not specifically reported were below their analytical detection limit (Table 2).

- Sample not analyzed or measured for these parameters.

\* Found below detection limit for analytical method.



# NAVAL CONSTRUCTION BATTALION CENTER Gulfport, Mississippi Installation Restoration Program

*The Installation Restoration (IR) program is an environmental program conducted nationwide by the Department of Defense to identify and address contamination from past practices which do not meet today's environmental standards. This fact sheet is the seventh in a series informing interested citizens about the IR program at NCBC Gulfport. Fact sheets will be produced at program milestones and in response to other items of public interest. Distribution is coordinated through the Public Affairs Office at NCBC Gulfport, telephone: (601) 871-2393.*

## FACT SHEET 7: Site 2, World War II Landfill

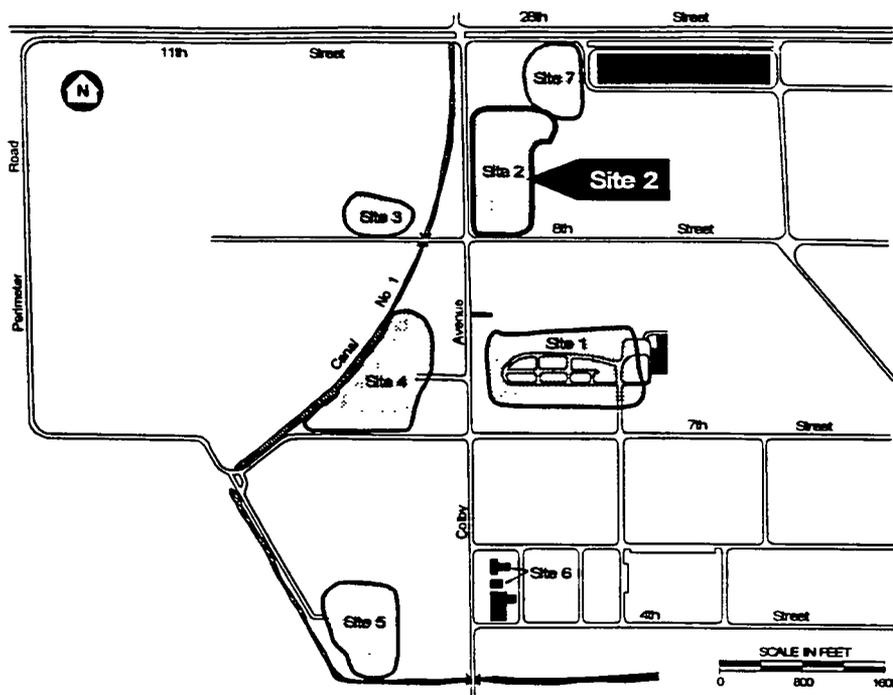


Exhibit 1. Site 2 is located on the northeast corner of the intersection of Colby Avenue and Eighth Street.

### DESCRIPTION OF SITE 2

Site 2 was operated as a landfill from 1942 to 1948. The 11-acre landfill received general trash from dumpsters located throughout the Seabee Center. Wastes were reportedly burned in the northern part of the site, then buried in 8-foot trenches located in the southern part of the site.

The majority of wastes in the landfill included general trash such as paper, cardboard, wood, and

garbage. In addition, some liquids, such as paints, paint thinners, solvents, oils, and fuels may have been placed into the landfill. Site 2 is now covered with pines and underbrush.

### PRIOR INVESTIGATIONS

**Initial Assessment Study (IAS):** The IAS was completed in 1985. The IAS included interviewing people who were knowledgeable

about activities at the base and reviewing records to determine if further environmental investigation was needed. The IAS recommended Site 2 for further study.

**Verification Study:** The Verification Study was completed in 1987. This study used specialized (geophysical) equipment to find the boundary of the landfill. In addition, the following samples were collected for laboratory analysis:

- two samples from groundwater wells and
- one surface water and sediment sample.

Chromium and lead were found in samples of the sediment and groundwater at Site 2. These metals are naturally occurring, and are, therefore, commonly found in environmental samples.

**Basewide Sampling:** A sampling program was performed throughout the entire base in December 1994. This program is further described in Exhibit 2.

Preliminary results of the groundwater samples taken in the vicinity of Site 2 found low levels of volatile and semivolatile organic compounds, metals, dioxins, and pesticides. All substances were found at concentrations below Federal standards.

These findings were reported immediately to the Mississippi Department of Environmental Quality and the Gulfport community. A technical evaluation of the results has not yet been completed to determine if these substances pose a health or environmental concern.

### WHAT'S NEXT FOR SITE 2?

The next typical step in the IR program process is to complete an in-depth environmental study, called a Remedial Investigation and Feasibility Study (often referred to as an RI/FS).

The Remedial Investigation includes collection and evaluation of environmental data. An assessment of potential ecological and human health effects of chemicals found through data collection is part of this evaluation. The Feasibility Study is an engineering evaluation of the best methods for cleaning up the site.

#### EXHIBIT 2. WHAT DID WE LOOK FOR IN THE BASEWIDE SAMPLING PROGRAM?

**Metals** include naturally occurring elements such as copper, arsenic, and lead. Household items that commonly contain metals include paint, batteries, coins, and electrical components.

**Herbicides** are chemicals used to kill unwanted plants and weeds. Common herbicides include Round-Up<sup>®</sup> and 2,4-D.

**Pesticides** are chemicals to eliminate insects and other pests. Flea collars, roach and ant killers, and household plant and garden sprays all contain pesticides.

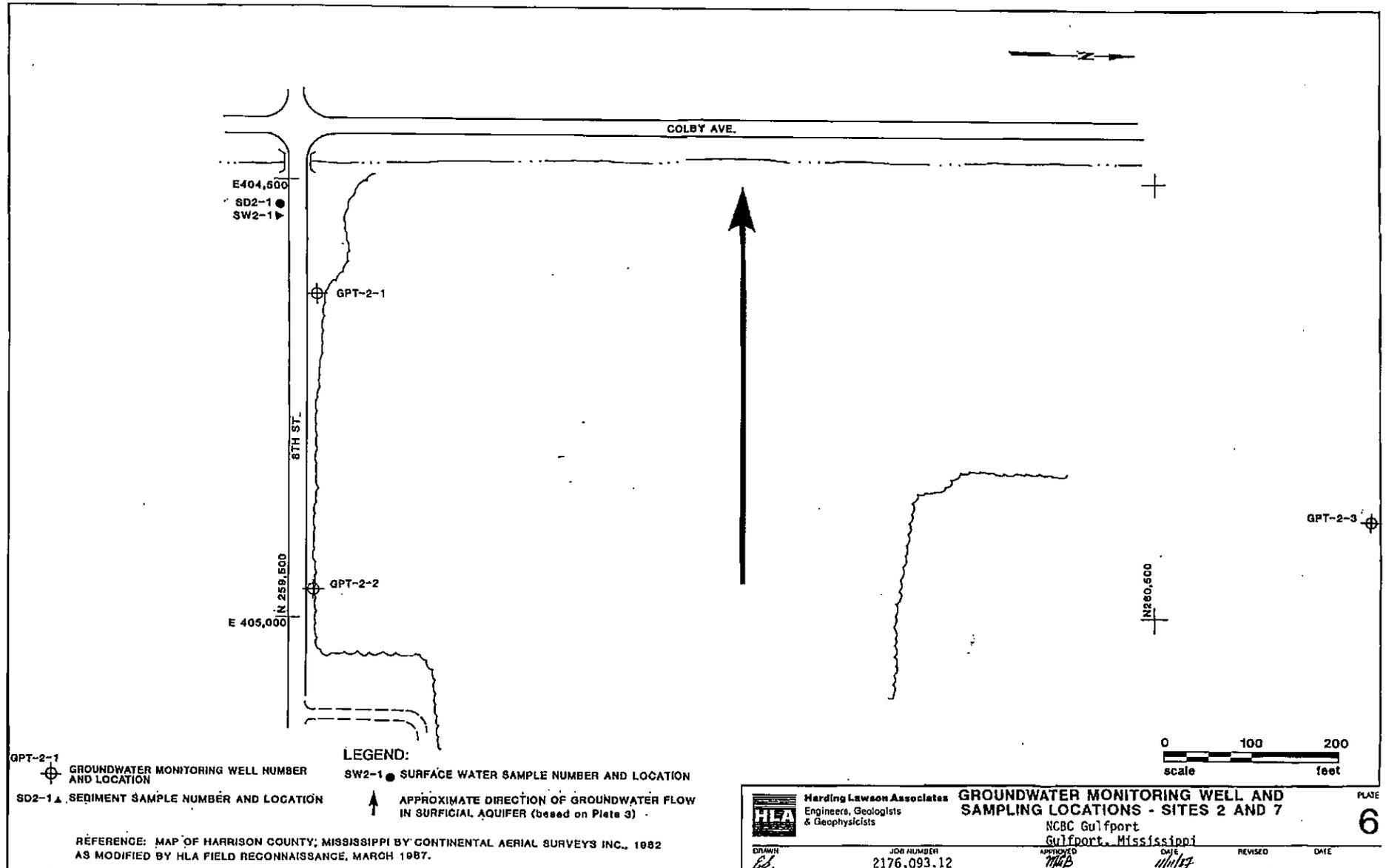
**Volatile organic compounds**, also known as VOCs, include solvents, paint thinner, and mineral spirits. Other household products that usually contain VOCs include hair spray, nail polish remover, and air fresheners. Common components of gasoline, such as benzene, toluene, and xylene, are VOCs.

**Semivolatile organic compounds**, also known as SVOCs, are a common component of asphalt, coal tar, and pitch. A good example of a naturally occurring SVOC is naphthalene, which is the main ingredient in many furniture refinishing products.

### ADDITIONAL INFORMATION

All reports discussed in this fact sheet are available at the NCBC Gulfport IR Program Information Repository located in the:

Gulfport Harrison County Library  
Reference Section  
21st Avenue (Highway 90)  
Gulfport, MS 39501  
Telephone: (601) 863-6411



**LEGEND:**

⊕ GPT-2-1 GROUNDWATER MONITORING WELL NUMBER AND LOCATION  
 ⊕ SW2-1 ● SURFACE WATER SAMPLE NUMBER AND LOCATION  
 ▲ SD2-1 ▲ SEDIMENT SAMPLE NUMBER AND LOCATION  
 ↑ APPROXIMATE DIRECTION OF GROUNDWATER FLOW IN SURFICIAL AQUIFER (based on Plate 3)

REFERENCE: MAP OF HARRISON COUNTY, MISSISSIPPI BY CONTINENTAL AERIAL SURVEYS INC., 1982 AS MODIFIED BY HLA FIELD RECONNAISSANCE, MARCH 1987.

	<b>Harding Lawson Associates</b> Engineers, Geologists & Geophysicists	<b>GROUNDWATER MONITORING WELL AND SAMPLING LOCATIONS - SITES 2 AND 7</b> NCBC Gulfport Gulfport, Mississippi	PLATE <b>6</b>
	DRAWN <i>Ed.</i>	JOB NUMBER 2176,093.12	APPROVED <i>THB</i>
		REVISED	DATE

## **APPENDIX C**

### **HUMAN HEALTH RISK ASSESSMENT METHODOLOGY AND ECOLOGICAL RISK ASSESSMENT METHODOLOGY**

# **APPENDIX C1**

## **HUMAN HEALTH RISK ASSESSMENT METHODOLOGY**

### **1.0 INTRODUCTION**

The human health risk assessment (HHRA) will be conducted to evaluate potential site-related risks to receptors associated with Site 2. This site is located on the Pine Bayou Golf Course in the northern portion of NCBC Gulfport. The site operated as a landfill from 1942 until 1948. During this time, nearly all of the solid waste and some liquid and chemical waste generated at the facility were disposed of at this site. The site is currently used as a fairway for the Pine Bayou Golf Course operated by NCBC Gulfport and a Presumptive Remedy, as prescribed in the USEPA guidance document (USEPA, 1993a), will be applied to the site. The Presumptive Remedy includes containment of the landfill contents and prevention of contaminant migration in the future.

The HHRA will be developed using the guidance documents published by U.S. Environmental Protection Agency (USEPA), USEPA Region 4 since Site 2 is being investigated pursuant to the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). The HHRA will be structured and reported according to the guidelines of the RAGS, Human Health Evaluation Manual, Part D: Standardized Planning, Reporting, and Review of Superfund Risk Assessments (RAGS Part D) (USEPA, 2001). The HHRA will consist of the following five components:

- Data evaluation
- Exposure assessment
- Toxicity assessment
- Risk characterization
- Uncertainty Analysis

### **2.0 DATA EVALUATION**

Data evaluation, the first component of a HHRA, is a media-specific task that begins with compilation of relevant analytical data and concludes with the selection of chemicals of potential concern (COPC) to be evaluated in the assessment. The data available for the Site 2 will be reviewed in terms of data quality; only validated data are used in the assessment. Environmental samples selected for HHRA will be

summarized in tables. A media-specific list of COPC will be selected based on a screening methodology (See Section 3.3 of this appendix).

Fixed-base analytical results (i.e., results from a fixed-base laboratory and not from field-analytical results) from field investigations for lists of target analytes will be used in the quantitative risk evaluation. Field measurements and data regarded as rejected (i.e., that were qualified as “R” during data validation) will not be used in the quantitative risk assessment. If a chemical was not detected in an environmental medium, but its reported detection limits (sample quantitation limits [SQLs]) for the environmental samples exceeds the COPC toxicity screening-levels, that chemical will be qualitatively discussed in the uncertainty analysis section.

### **3.0 EXPOSURE ASSESSMENT METHODOLOGY**

The exposure assessment component of the HHRA will include an updated conceptual site model (CSM) presented in Figure 3 of this UFP SAP. The CSM will identify the exposure pathways by which human receptors may come in contact with environmental media within the Site 2 (or downgradient of the Site 2) after the Presumptive Remedy is completed. The CSM will depict the relationships among the following elements of a *complete* exposure pathway; i.e., a pathway that potentially results in human exposure and is evaluated (qualitatively or quantitatively) in an HHRA:

- Site 2 sources of contamination
- Contaminant release mechanisms and transport/migration pathways
- Chemicals of potential concern
- Exposure routes
- Potential receptors

#### **3.1 SITE 2 SOURCES OF CONTAMINATION**

The primary source of contamination at Site 2 is the refuse that was disposed when the site was used as an active landfill. The majority of the waste disposed at the site was general refuse and inert material such as paper, cardboard, wood, and household garbage. Liquid waste such as paints, paint thinners, solvents, oils, and fuels were also reportedly disposed at the site (incinerated or buried). There is no documentation indicating the exact volume of waste that was disposed at the site. Paints used at NCBC Gulfport during the time Site 2 was operational typically contained cadmium, lead, and chromium. These metals, as well as products of incomplete combustion and dioxins/furans formed during combustion, may exist at the site. Chemical constituents associated with these types of wastes include metals, VOCs, SVOCs, pesticides, herbicides, and PCBs.

### **3.2 CONTAMINANT RELEASE MECHANISMS AND TRANSPORT/MIGRATION PATHWAYS**

Currently Site 2 is the fairway of a golf course and the source of contamination is covered by fill. This source of contamination will be capped after the Presumptive Remedy is installed. These two conditions are discussed in the next two subsections.

#### **3.2.1 Pre Presumptive Remedy Conditions**

Site 2 is currently covered with fill that ranges in thickness from 6 inches to over 2 feet as a result of golf course construction. This cover may somewhat limit infiltration of stormwater into the subsurface during precipitation events. Because waste material may be present in the subsurface, subsurface soil may be contaminated and groundwater that comes in contact with the waste may also become contaminated. As the groundwater migrates through the site, downgradient subsurface soil and groundwater may also be impacted. Groundwater flow is most likely towards the pond on the eastern side of the site; however, there is a potential groundwater divide that may cause groundwater to flow to the west as well. In this case, groundwater may also discharge into the western ditch.

Surface water runoff from rainfall events at Site 2 flows toward the pond on the eastern side of the site and toward the ditch to the west (Figure 2 in the USP SAP). This water is then conveyed toward storm water Outfalls 1 and 3 on the northern end of the site (Figure 5 in the USP SAP). There is little likelihood of contaminated media (i.e., contaminated surface soil) transfer due to surface water runoff from rainfall events since Site 2 is covered with fill and the fairways are routinely managed to maintain a healthy grass cover.

#### **3.2.2 Post Presumptive Remedy Conditions**

After the presumptive remedy is constructed there should be no intermedia transfer (waste leachate to subsurface soils to groundwater) due to infiltration following rainfall events. In addition there should be no contaminated media transfer from surface soils to ditches during or from to surface water runoff from rainfall events.

### **3.3 CHEMICALS OF POTENTIAL CONCERN**

Media-specific list of chemicals of potential concern (COPC) will be selected for subsurface soils and groundwater. A screening level methodology for selecting these COPCs will be used to develop the list of COPCs for subsurface soils and groundwater. The screening level methodology uses peer reviewed generic clean-up criteria to screen in chemicals for further evaluation. The screened-in chemicals become the COPCs. The screening level methodology is presented in the next two subsections.

### **3.3.1 Subsurface COPCs**

The list of COPCs, associated with subsurface soils, will be based on comparing the maximum detected concentration against the lower of the USEPA Regional Screening Level (RSL) for direct contact (RSL-dc), the RSL for Migration to Groundwater Soil (i.e. RSL-leachability) or the Mississippi Department Of Environmental Quality (MDEQ) Unrestricted Soil Tier 1 Target Remediation Goals (Unrestricted Tier 1TRGs). The threshold for this comparison will correspond to a systemic hazard quotient of 0.1 for noncarcinogens or an incremental lifetime cancer-risk of  $1 \times 10^{-6}$  for carcinogens. The RSLs were developed by the U.S. Department of Energy's Oak Ridge National Laboratory (USEPA, 2010). The Tier 1 TRGs were developed by the MDEQ (MDEQ, 2002).

Analytes with soil concentrations that exceed the screening criteria and the background concentration (if available ) will be retained as soil COPCs.

### **3.3.2 Groundwater COPCs**

The list of COPCs, associated with groundwater, will be based on two thresholds. Under the first threshold, the the maximum detected concentration will be compared against the minimum of the Groundwater Tier 1 TRG (MDEQ, 2002), the Maximum Contaminant Level (MCLs) Safe Drinking Water Act or the USEPA Tapwater RSL (USEPA, 2010). Under the second threshold, the maximum detected concentration of volatile organic compounds (VOCs) will be compared against calculated soil vapor screening values. These screening values will be calculated according to the methodology present in Appendix D of USEPA's Draft Guidance for Evaluating the Vapor Intrusion into Indoor Air from Groundwater and Soils (USEPA, 2002) and Appendix A of the Department of Defense's DoD's Vapor Intrusion Handbook (DoD, 2009).

Analytes with groundwater concentrations that exceed the screening criteria and the background concentration (if available ) will be retained as ground water COPCs.

### **3.3.3 Decision Rules for Establishing COPC**

The following decision rules will be used to select COPC:

- As noted in Sections 3.3.1 and 3.3.2, a chemical detected in Site 2 media will be selected as a COPC for the HHRA if the maximum detected chemical concentration exceeds its respective

screening level and the chemical is present in the environmental media at concentrations greater than background levels.

- Essential nutrients will not be selected as COPC. USEPA guidance (USEPA, 1989) states that “Chemicals that are (1) essential human nutrients, (2) present at low concentrations (i.e., only slightly elevated above natural occurring levels), and (3) toxic at very high doses (i.e., much higher than those that could be associated with contact at the site) need not be considered further in the quantitative risk assessment.” Examples of such chemicals are iron, magnesium, calcium, potassium, and sodium.
- Surrogate COPC screening levels may be used for some chemicals. For example, in the past, risk-based COPC screening levels were not available for some chemicals [e.g., acenaphthylene, benzo(g,h,i)perylene, phenanthrene] media due to lack of toxicity criteria. Currently, MDEQ Unrestricted Tier 1TRGs are available for these three chemicals.

Chemicals without COPC screening levels or appropriate surrogate-chemical COPC screening levels will be evaluated qualitatively in the COPC selection section and/or in the uncertainty section of the HHRA. The evaluation will consider the number of times the chemical was detected and the magnitudes of the observed concentrations.

### **3.4 EXPOSURE-POINT CONCENTRATIONS**

Exposure-point concentrations (EPCs) are the concentrations to which receptors are assumed to be exposed. The EPCs will be calculated for COPCs only for each exposure unit (EU) identified for the Site 2. An EU is the area over which receptor activity is expected. EUs will be determined based on the spatial distribution of the data and nature of receptor activities.

The following guidelines will be used to calculate EPCs for COPC concentrations for each EU:

- For soil and groundwater data sets containing at least five samples, the 95% upper confidence limit (UCL) on the arithmetic mean, which is based on the distribution of the data set, will be selected as the EPC unless the UCL value exceeds the maximum detected concentration. In this case, the maximum detected concentration will be used as the EPC. The maximum concentration will also be used as the EPC in the event of an insufficient number of detections to calculate a 95% UCL (i.e., less than four positive detections in a data set) in accordance with USEPA guidance (USEPA, 2011). EPCs will be calculated following USEPA's Calculating Upper Confidence Limits for Exposure-Point Concentrations at Hazardous Waste Sites (USEPA,2002) and using USEPA's ProUCL software (USEPA, 2011).

- The sample quantitation limit will be used as an input for non-detects in USEPA's ProUCL software to calculate the 95% UCL in accordance with ProUCL guidance (USEPA, 2011). Duplicates will be averaged to calculate the EPCs for COPC in all media within the Site 2.

### **3.5 POTENTIAL RECEPTORS**

Potential human receptors, currently at Site 2, include people employed at NCBC Gulfport, trespassers, construction workers, and recreational site users. However, unless potentially contaminated wastes/subsurface soils are excavated and distributed atop surface soils, construction workers are the only receptors likely to contact contaminated subsurface wastes/soils. Trespassers, recreational users, and base personnel are unlikely to contact subsurface wastes/soils or groundwater under current site conditions and land use.

It is anticipated that land use controls (LUCs) will become part of the risk mitigation methods used after the Presumptive Remedy is constructed. Therefore it is unlikely that in the foreseeable future, children or adults will be living on Site 2. However land use may change; therefore the future child and adult resident are included as potential receptors.

Risks to the following potential receptors will be evaluated:

#### **3.5.1 On-site Construction Workers**

Construction workers are plausible on-site adult receptors who could be exposed to chemicals in subsurface soil through incidental ingestion and dermal contact and through inhalation of airborne contaminants emanating from soil. Construction workers could also be exposed to chemicals in shallow groundwater pooling in an excavation ditch through dermal contact and through inhalation of airborne contaminants; however, construction workers are not expected to ingest groundwater.

#### **3.5.2 Future Child and Adult On-site Residents**

The hypothetical future, on-site residential land use scenario will be evaluated in the HHRA to facilitate risk management decisions. The HHRA will assume that a future, on-site resident may be exposed to the subsurface soils through ingestion, dermal contact, and inhalation of chemicals emitted from soil to air since future construction activities could potentially redistribute subsurface soil to the surface. Additionally, future on-site residents could hypothetically be exposed to chemicals in groundwater through ingestion (i.e., drinking water), dermal contact, and inhalation of VOCs if the groundwater resource were to be used for domestic purposes. The results of the HHRA will be used to assist the risk managers in

determining the need for deed restrictions to prohibit domestic use of the groundwater resource and/or to prohibit the development of the property for residential purposes and/or the excavation of contaminated subsurface soils. (Please note that the HHRA will not include risk estimates for a base personnel, trespassers, or recreational users hypothetically exposed to contaminated subsurface soils at Site 2 because: 1) A presumptive remedy has already been determined for the site, 2) Any risk estimates calculated for the hypothetical future resident exposed to contaminated subsurface soils will exceed those estimated for base personnel, trespassers, or recreational users (i.e., risk estimates for the hypothetical future resident will support the evaluation of the need for deed restrictions to prohibit the development of the property or excavation of contaminated subsurface soils). The list of receptors and exposure pathways evaluated may be further expanded to include hypothetical on-site or off-site workers or residents exposed via the vapor intrusion pathway (working/residing in buildings) if VOC concentrations in the groundwater exceed the vapor intrusion screening levels referenced above. If off-site groundwater concentrations exceed groundwater screening levels referenced above, the evaluation may also be expanded to evaluate the hypothetical future use of off-site groundwater for domestic purposes. Finally, the HHRA may be expanded to include receptor (hypothetical recreational user, resident) exposure (incidental ingestion, dermal contact) to surface waters and sediments if chemical concentrations detected in these media exceed the referenced RSL-based COPC screening levels listed in the preceding narrative by a factor of 10.

### **3.6 ESTIMATING CHEMICAL INTAKES**

Intakes for the identified potential receptor groups will be calculated using current USEPA risk assessment guidance and based on the concept of a “reasonable maximum exposure” (USEPA, 1989). The estimated chemical intakes will be presented in the risk assessment spreadsheets (using the USEPA RAGS Part D Tables format). Standard chemical-intake assumptions and equations presented in USEPA guidance will be used to calculate chemical intakes for soil (ingestion, dermal contact, and inhalation) and groundwater (ingestion, dermal contact, and inhalation).

The USEPA’s Supplemental Guidance for Assessing Susceptibility from Early Life Exposure to Carcinogens (USEPA, 2005) recommends adjusting for the toxicity of carcinogenic chemicals that act mutagenically when evaluating early life exposures to contaminants. The guidance recommends using age-dependent adjustment factors (ADAFs) in concert with age-specific exposure estimates when assessing cancer risks. For absent chemical-specific data, the supplemental guidance recommends the default adjustments, which reflect that cancer risks are generally higher from early-life exposures than from similar exposures later in life. These adjustments will be applied using the same method as that used by USEPA to develop the RSLs. This approach will be used only for chemicals identified as mutagenic in the USEPA RSL screening table; e.g., carcinogenic polycyclic aromatic hydrocarbons (cPAHs).

## 4.0 TOXICITY ASSESSMENT

The reference dose (RfD) is the toxicity value used to evaluate noncarcinogenic health effects for ingestion and dermal exposures. The reference concentration (RfC) is used to evaluate noncarcinogenic health effects for inhalation exposures. The RfD and RfC estimate a daily exposure level for a human population that is unlikely to pose an appreciable risk during a portion or for all of a human lifetime. Carcinogenic effects are quantified using the cancer slope factor (CSF) for ingestion and dermal exposures and using inhalation unit risks (IUR) for inhalation exposure that are plausible upper-bound estimates of the probability of the development of cancer per unit intake of the chemical over a lifetime.

### 4.1 TOXICITY CRITERIA FOR ORAL AND INHALATION EXPOSURES

Oral RfDs and CSFs and inhalation RfCs and IURs used in the risk assessment will be obtained from the following primary USEPA literature sources (USEPA, 2003):

- IRIS — USEPA's "Integrated Risk Information System" online database (this is the preferred source of toxicity values).
- USEPA's Provisional Peer Reviewed Toxicity Values (PPRTVs) — USEPA's Office of Research and Development/National Center for Environmental Assessment (NCEA) Superfund Health Risk Technical Support Center develops chemical-specific PPRTVs when requested by USEPA's Superfund program.
- Other toxicity values — These sources include, but are not limited to, California Environmental Protection Agency (Cal/EPA) toxicity values, federal Agency for Toxic Substances and Disease Registry (ATSDR) values, and the Annual Health-Effects Assessment Summary Tables (HEAST) (USEPA, 1997).

#### 4.1.1 Toxicity Criteria for Dermal Exposure

RfDs and CSFs in the scientific literature are typically expressed as "administered" (i.e., not absorbed) doses. Oral dose-response parameters based on administered doses must be adjusted to absorbed doses before they can be compared to estimated dermal exposure intakes. When oral absorption is essentially complete (i.e., 100%), an absorbed dose is equivalent to the administered dose, and therefore no toxicity adjustment is necessary. Conversely, when the gastrointestinal absorption of a chemical is poor (e.g., 1%), the absorbed dose is smaller than the administered dose; thus, toxicity factors based on absorbed dose should be adjusted to account for the difference in the absorbed dose relative to the administered dose. USEPA (2004) recommends a 50% absorption cut-off to reflect the intrinsic variability

in analyzing absorption studies. Therefore, the adjustment from administered to absorbed dose will only be performed when the chemical-specific gastrointestinal absorption efficiency was less than 50%.

#### 4.1.2 Toxicity Criteria for the Carcinogenic Effects of cPAHs

The toxic effects of these chemicals will be evaluated using toxicity-equivalency factors (TEFs) based on the potency of each compound relative to that of benzo(a)pyrene, as presented in current USEPA guidance (USEPA, 1993). TEFs are used to convert each individual cPAH concentration into an equivalent concentration of benzo(a)pyrene.

## 5.0 RISK CHARACTERIZATION

Quantitative estimates of risk for chemicals will be calculated according to risk assessment methods outlined in USEPA guidance (USEPA, 1989) and updates to the guidance (e.g., RAGS Part F). Lifetime cancer risks will be expressed in the form of dimensionless probabilities referred to as incremental lifetime cancer risks (ILCRs), based on CSFs and IURs. Noncarcinogenic risk estimates will be presented in the form of hazard quotients (HQs) and hazard indices (HIs), which are determined by comparing intakes against published RfDs and RfCs.

As points of reference, USEPA defines the range of  $1 \times 10^{-4}$  to  $1 \times 10^{-6}$  as the ILCR target range for hazardous waste facilities addressed under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the Resource Conservation and Recovery Act (RCRA). Individual or cumulative ILCRs greater than  $1 \times 10^{-4}$  are generally considered “unacceptable” by USEPA. Risk management decisions are necessary when the ILCR is within  $1 \times 10^{-4}$  to  $1 \times 10^{-6}$ . USEPA typically does not require remediation when the cumulative ILCR is less than  $1 \times 10^{-6}$ .

An HI exceeding unity (1.0) indicates that noncarcinogenic health risks may be associated with exposure. If an HI exceeds unity, target-organ effects associated with exposure to COPC are considered. Only those HQs for chemicals affecting the same target organ(s) or exhibiting similar critical effect(s) are regarded as truly additive. Consequently, the cumulative HI could exceed 1.0, but no adverse health effects would be anticipated unless the COPC affected the same target organ or exhibited the same critical effect (i.e., unless target-organ-/critical-effect-specific HIs exceeded 1).

As a general guideline, a “no further action” recommendation will be proposed whenever the cancer risk estimates and total HIs (estimated on a target-organ/target-effect basis) for receptors of concern are less than  $1 \times 10^{-4}$  and 1, respectively. Otherwise, in most cases, the need for further action (most likely deed restrictions/institutional controls) will need to be further evaluated. However, the  $1 \times 10^{-4}$  risk benchmark

should not be viewed as a discrete limit. Risks slightly greater than  $1 \times 10^{-4}$  may be considered “acceptable” (i.e., protective) if justified by site-specific conditions, including any uncertainties about the nature and extent of contamination and associated risks. The following factors will be considered in this determination:

- The magnitude of the media-specific risk estimates.
- Significant uncertainties in the baseline HHRA that would overestimate baseline risk assessment results.
- Significant uncertainties in EPC estimates that would overestimate baseline risk assessment results.

## **6.0 UNCERTAINTY ANALYSIS**

The uncertainty analysis component of the HHRA will provide a summary of uncertainties inherent in the risk assessment and include a discussion of how they may affect the quantitative risk estimates and conclusions of the risk analysis.

## **7.0 REFERENCES**

DoD 2009, DoD Vapor Intrusion Handbook. Prepared by the Tri-Service Environmental Risk Assessment Workgroup. January 2009

MDEQ, 2002. Risk Evaluation Procedures for Voluntary Cleanup and Redevelopment of Brownfield Sites, Tier I Evaluation Target Risk Level. February

USEPA (U.S. Environmental Protection Agency), 1989. Risk Assessment Guidance for Superfund (RAGS), Volume I. Human Health Evaluation Manual, Part A. Interim Final. December

USEPA (U.S. Environmental Protection Agency), 2002. Calculating Upper Confidence Limits for Exposure Point Concentrations at Hazardous Waste Sites. OSWER 9285.6 10. Office of Emergency and Remedial Response, Washington, D.C., December.

USEPA, 1993a. Presumptive Remedy for CERCLA Municipal Landfill Sites. Office Emergency and Remedial Response Hazardous Site Control Division 5203G Quick Reference Fact Sheet; Directive: 9355.0-49FS; USEPA 540-F-93-035; PB 93-963339. September.

USEPA (U.S. Environmental Protection Agency), 1993. Provisional Guidance for Quantitative Risk Assessment for Polycyclic Aromatic Hydrocarbons. Office of Research and Development. Washington, D.C. EPA/600/R 93 089. July

USEPA (U.S. Environmental Protection Agency), 1997 U.S. EPA. Health Effects Assessment Summary Tables (HEAST). U.S. Environmental Protection Agency, Washington, D.C., 1997. <http://cfpub.epa.gov/ncea/cfm/recordisplay.cfm?deid=2877#Download>

USEPA (U.S. Environmental Protection Agency), 2001. Risk Assessment for Superfund (RAGS), Human Health Evaluation Manual, Part D: "Standardized Planning, Reporting, and Review of Superfund Risk Assessments" (RAGS Part D), Publication 9285.7 01D. December

USEPA, 2002. OSWER Draft Guidance for Evaluating the Vapor Intrusion into Indoor Air from Groundwater and Soils (Subsurface Vapor Intrusion Guidance). Office of Solid Waste and Emergency Response. EPA 530-D-02-004. November 2002.

USEPA (U.S. Environmental Protection Agency), 2003. Human-Health Toxicity Values in Superfund Risk Assessments. Office of Superfund Remediation and Technology Innovation, OSWER 9285.7-53, Washington, D.C. December.

USEPA (U.S. Environmental Protection Agency), 2004. Risk Assessment Guidance for Superfund: Volume I, Human-Health Evaluation Manual, Part E, "Supplemental Guidance for Dermal Risk Assessment." Office of Emergency and Remedial Response, Washington, D.C. July

USEPA (U.S. Environmental Protection Agency), 2005. Supplemental Guidance on Assessing Susceptibility from Early Life Exposure to Carcinogens. EPA/630/R 03/003F. Risk Assessment Forum, Washington, D.C. March.

USEPA (U.S. Environmental Protection Agency), 2010. Regional Screening Levels for Chemical Contaminants at Superfund Sites, prepared by Oak Ridge National Laboratory. [http://www.epa.gov/reg3hwmd/risk/human/rb-concentration\\_table/index.htm](http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm)

USEPA (U.S. Environmental Protection Agency), 2011. ProUCL Version 4.00.05 User Guide. Office of Research and Development, Washington, D.C. EPA/600/R 07/038. March 2011. <http://www.epa.gov/esd/tsc/software.htm>

## ECOLOGICAL RISK ASSESSMENT METHODOLOGY

The ecological risk assessment (ERA) will be conducted to evaluate potential site-related risks to ecological receptors at Site 2. The ERA will consist of Steps 1 through 3a of USEPA's 8-step ecological risk assessment process, and will be conducted in accordance with USEPA and Navy guidance (USEPA, 1997; 2000a; 2001; DON, 1999). Steps 1 through 3a consist of the following:

- Step 1            Screening-Level Problem Formulation and Ecological Effects Evaluation
- Step 2            Screening-Level Exposure Estimate and Risk Calculation
- Step 3a          Refinement of Preliminary Chemicals of Potential Concern

### **Problem Formulation**

Site 2 is located on the Pine Bayou Golf Course in the northern portion of NCBC Gulfport. Ecological habitat at the site consists largely of mowed grass. The golf course is subjected to intensive management practices in order to sustain the conditions desired by golfers. Soil invertebrates undoubtedly exist there, although invertebrate populations are presumably impacted by the current and historical use of insecticides at the golf course. The lack of concealing vegetation on the golf course, combined with the relative scarcity of invertebrates due to the recurring use of insecticides, results in a habitat type that is probably infrequently used by wildlife. Nevertheless, various bird species forage in the fairway and rough areas, consuming invertebrates in the soil and grass as well as seeds blown in from nearby wooded areas. The grass height, being similar to that in a recently mowed lawn, is too low to provide cover for small mammals such as shrews and mice. With the exception of invertebrates and birds, probably few receptors forage in the mowed grass during daylight hours. The site is undoubtedly traversed by some wildlife species, especially at night. In addition, a few areas of trees and shrubby vegetation are located within the site; wildlife such as birds and mammals would use these areas to some extent. A pond lies along the east edge of Site 2. Various species of fish presumably inhabit the pond, and wading birds such as herons and egrets probably forage there.

The former landfill and the original contaminant sources are covered from 6 inches to 6 feet of fill. Therefore, exposure to landfill-related contaminants by terrestrial ecological receptors is probably negligible. However, the possibility that soil has been disturbed during excavation, such as laying pipes or other activities, cannot be ruled out. As a conservative measure, therefore, it will be assumed that the soil cover is not of uniform thickness, and the soil exposure pathway will be assumed to be complete. To the extent that this is true, soil invertebrates such as earthworms and larval insects could be exposed to soil contaminants as they move through the soil and ingest soil particles while searching for food. Plants

are exposed to contaminants through direct contact as contaminants are absorbed through the roots and are then translocated to different parts of the plants (e.g., leaves, seeds).

Inhalation of particulates by mammals and birds will not be considered a complete pathway because there are no activities causing air contamination. Also, inhalation pathways are not typically evaluated in ERAs because of the uncertainty inherent in estimating exposure levels and toxicological effects. Therefore, the air inhalation pathway will not be evaluated in the ERA.

Aquatic organisms such as fish, and benthic organisms (i.e., invertebrate organisms that live on or in sediment) could be exposed to sediment and surface water contaminants through ingestion and direct contact. Higher trophic level animals such as birds and mammals that forage in the pond east of Site 2 can be exposed to site-related contamination through ingestion of contaminated food items and water. These animals may also incidentally ingest contaminants in sediment while preening feathers or feeding on items to which sediment has adhered. Absorption of contaminants from the gastrointestinal tract is the primary pathway of intake for upper trophic level receptors.

In summary, complete exposure pathways and routes of entry into biota at Site 2 consist of:

- direct contact with soil, sediment, and surface water
- ingestion of soil, sediment, and surface water
- Ingestion of contaminated food items by upper trophic level animals foraging in surface water.

An assessment endpoint is “an explicit expression of the environmental value that is to be protected,” while a measurement endpoint is “a measurable ecological characteristic that is related to the valued characteristic chosen as the assessment endpoint” (USEPA, 1997). Measurement endpoints represent the assessment endpoints chosen for a site, and are measures of biological effects (USEPA, 1997). USEPA Region 4 has specified that assessment endpoints for the screening-level assessment should be broad and generic. Therefore, the preliminary assessment endpoint for Site 2 will be the protection of terrestrial, benthic, and aquatic biota from adverse effects of chemicals on their growth, survival, and reproduction. The preliminary measurement endpoints are chemical concentrations in surface soil, sediment, and surface water that are associated with no adverse effects on growth, survival, and reproduction of terrestrial, benthic, and aquatic organisms. The measurement endpoints are represented by ecological screening values (ESVs) for surface soil, sediment, and surface water.

The ESVs are based on conservative endpoints and sensitive ecological effects data, and thus, the screening values represent chemical concentrations associated with a low probability of unacceptable risks to ecological receptors. For this reason, USEPA Region 4 considers the screening values to be protective of invertebrates, plants, and aquatic organisms as well as upper level receptors such as birds

and mammals. In the screening level ERA, therefore, a distinction is not made between measurement endpoints associated with direct toxicity versus measurement endpoints associated with food-chain effects.

### **Screening-Level Ecological Effects Evaluation**

Soil screening values used in the screening level ERA will be USEPA Ecological Soil Screening Levels (Eco-SSLs) and ESVs established by USEPA Region 4 (USEPA, 2001). If an Eco-SSL is available for a given chemical, the lowest Eco-SSL among plant, invertebrate, mammal, and avian values will be used as the screening value. Eco-SSLs will be preferentially used as soil screening values, but Eco-SSLs are currently available for only a few chemicals. USEPA Region 4 ESVs (USEPA, 2001) will be used as screening values for chemicals that do not have an Eco-SSL. The term "soil ESV" will be generally used for brevity in the ERA to refer to either the Eco-SSL or the Region 4 soil ESV.

ESVs for surface water and sediment will be those established by USEPA Region 4 (USEPA, 2001).

### **Screening-Level Risk Calculation**

The screening level risk calculation step compares maximum concentrations of chemicals in surface soil, sediment, and surface water to ESVs. The ratio of the maximum concentration to the ESV is called the screening hazard quotient (HQ). Analytes with maximum concentrations less than or equal to ESVs ( $HQ \leq 1$ ) will be dropped from further consideration, while those that exceed ESVs ( $HQ > 1$ ), or do not have ESVs, will be retained as ecological chemicals of potential concern (COPCs). An HQ value greater than 1 indicates that ecological receptors are potentially at risk, and further evaluation or additional data may be necessary to confirm with greater certainty whether ecological receptors are actually at risk, especially since most toxicity benchmarks are developed using conservative exposure assumptions. Chemicals that were retained as COPCs will be evaluated in Step 3A so that risk managers can determine if further investigation is warranted.

Calcium, magnesium, potassium, and sodium will not be considered to be COPCs because they are essential nutrients that can be tolerated by living systems even at relatively high concentrations. There have been no activities at NCBC Gulfport that have resulted in known releases of high levels of these four chemicals at Site 2.

## **Refinement of Preliminary Chemicals of Potential Concern**

At this point, the first two steps of the ERA will have been completed. The ERA process includes a series of scientific/management decision points (SMDPs) (USEPA, 1997). The first SMDP occurs at the end of Step 2, and requires the risk managers to evaluate and approve or redirect the work up to that point and determine whether the risk assessment will continue into Step 3. However, USEPA Region 4 recognizes that most ERAs will proceed into Step 3, and facilities are encouraged to submit the results of Steps 1-3 as a single deliverable document (USEPA, 2000a). With this in mind, if the screening level ERA indicates a potential for adverse effects, a more thorough assessment will be warranted. If so, the risk assessment process for the site will proceed into Step 3 (Baseline Risk Assessment Problem Formulation).

The baseline ERA assessment begins with a more balanced evaluation of the conservativeness inherent in the first two steps of the risk assessment process (USEPA, 1997; DON, 1999). The initial phase of Step 3 is typically known as Step 3a, and consists of a refinement of the conservative exposure assumptions in order to more realistically estimate potential risks to plants, invertebrates, and wildlife receptors. Examples of factors typically considered during Step 3a include toxicological evaluation of COPCs, spatial distribution of contaminants, frequency of detection, and habitat quality (USEPA, 1997; DON, 1999). Furthermore, the preliminary assessment and measurement endpoints are refined, the site conceptual model is developed, and initial food-chain modeling is conducted (at sites where applicable) to evaluate risks to upper level receptors (USEPA, 2000a). The objective of the Step 3a refinement is to better define those chemicals that contribute to potentially unacceptable levels of ecological risk, and to identify and eliminate from further consideration those chemicals that are initially selected as COPCs because of the use of very conservative assumptions.

Based on the habitats present at Site 2, and the migration pathways and routes of exposure of chemicals at the site, the site-specific assessment endpoints to be evaluated in the Refinement of Preliminary Chemicals of Potential Concern phase are the protection of the following groups of receptors from adverse effects of site-related contaminants on growth, survival, and reproduction:

- soil invertebrates
- terrestrial plants
- benthic invertebrates
- aquatic organisms
- piscivorous birds
- piscivorous mammals

The ERA will present details supporting the selection of the above assessment endpoints.

Adverse impacts on survival, growth, and reproduction of plants, soil invertebrates, benthic invertebrates, and aquatic organisms will be evaluated by comparing chemical concentrations in surface soil, sediment, and surface water to USEPA Eco-SSLs and USEPA Region 4 ESVs and to other pertinent guidelines. Adverse impacts on survival, growth, and reproduction of piscivorous birds and mammals will be evaluated by comparing estimated ingested doses of contaminants in surface water, sediment, and food items to oral toxicity threshold values.

The term “piscivorous” will be used in a broad sense to describe birds and mammals that prey upon not only fish, but on a variety of aquatic and sediment dwelling organisms (e.g., crayfish, frogs). Piscivorous birds that forage in water bodies near Site 2 include wading birds such as herons and egrets. Piscivorous mammals presumed to be present include the river otter and mink. The raccoon is often thought of as piscivorous, and it does consume aquatic organisms, but the majority of its diet typically consists of non-aquatic animal and plant tissues (USEPA, 1993). Piscivorous birds and mammals can be exposed to and accumulate site-related contaminants that have accumulated in prey items obtained from the site. This would be especially applicable for contaminants such as PCBs, organochlorine pesticides, and certain metals.

The mink will be used to represent piscivorous mammals and the green heron will be used to represent piscivorous birds. These species have a high probability of exposure to surface water and sediment contaminants based on their diet and habitat preferences.

As mentioned earlier, the former landfill is covered by fill dirt within an intensively managed golf course fairway. With this in mind, the surface soil exposure pathway for terrestrial receptors such as birds, mammals, and reptiles is probably negligible and insignificant.

Food-chain modeling will be conducted to evaluate potential risks to representative piscivorous receptors from screening-level COPCs in Site 2 sediment and surface water that are known to bioaccumulate or biomagnify. USEPA Region 4 considers chemicals in this category to consist of those so designated in *Bioaccumulation Testing and Interpretation for the Purpose of Sediment Quality Assessment, Status and Needs* (USEPA, 2000b), with the exception of PAHs. USEPA Region 4 does not consider PAHs to bioaccumulate unless they are present at percent levels in soil or sediment.

Risk via the food chain will be evaluated using two scenarios. The first scenario will use maximum detected COPC concentrations in sediment and surface water and conservative assumptions for body weight, food consumption, and sediment ingestion. The second scenario will use average COPC concentrations, and less conservative values for body weight, food consumption, and sediment ingestion.

The ERA will contain details regarding all exposure factors (body weight, food consumption, sediment ingestion, etc). For each scenario, ingested doses for birds and mammals will be calculated using the equation shown below.

$$PD = [(C_{\text{food}} \times I_f) + (C_{\text{water}} \times I_w) + (C_{\text{sed}} \times I_{\text{sed}})] \times \text{AUF} / \text{BW} \quad (\text{Equation 1})$$

where: PD = predicted dose from the ingestion of food and water and the incidental ingestion of sediment (mg/kg/day)

$C_{\text{food}}$  = contaminant concentration in food (mg/kg)

$I_f$  = food ingestion rate (kg/day)

$C_{\text{water}}$  = contaminant concentration in water (mg/L)

$I_w$  = water ingestion rate (L/day)

$C_{\text{sed}}$  = contaminant concentration in sediment (mg/kg)

$I_{\text{sed}}$  = sediment ingestion rate (kg/day)

AUF = area use factor (portion of home range that overlaps Site 2)

BW = weight of receptor (kg)

Risk to piscivorous receptors as a result of exposure to COPCs in surface water and sediment will be determined by comparing the predicted dose to toxicity reference values (TRVs) representing acceptable daily doses in mg/kg-day. The TRVs will be developed from no observed adverse effect levels (NOAELs) and lowest observable adverse effect levels (LOAELs) obtained from toxicity studies. The TRVs to be used in the food-chain model will come from the ORNL Toxicological Benchmarks for Wildlife: 1996 Revision (Sample, et al., 1996), EPA Eco-SSLs, and other sources as necessary.

## REFERENCES

DON (Department of the Navy). 1999. Navy Policy for Conducting Ecological Risk Assessments. Memo from Chief of Naval Operations to Commander, Naval Facilities Engineering Command, 05 April 1999. Department of the Navy, Washington, DC.

Sample, B.E., D.M. Opresko, and G.W. Suter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Oak Ridge National Laboratory. June. ES/ER/TM-86/R3.

USEPA (U.S. Environmental Protection Agency), 1993. Wildlife Exposure Factors Handbook. Office of Research and Development. Washington, D.C. EPA/600/R-93/187a. December.

USEPA (U.S. Environmental Protection Agency), 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments. Interim Final. Environmental Response Team. June 5.

USEPA (U.S. Environmental Protection Agency), 2000a. Region 4 Amended Guidance on Ecological Risk Assessment at Military Bases: Process Considerations, Timing of Activities, and Inclusion of Stakeholders. June 23.

USEPA (U.S. Environmental Protection Agency), 2000b. Bioaccumulation Testing and Interpretation for the Purpose of Sediment Quality Assessment, Status and Needs. Office of Water, Office of Solid Waste. EPA 823-R-00-001. February.

USEPA (U.S. Environmental Protection Agency), 2001. Region 4 Ecological Risk Assessment Bulletins – Supplement to RAGS. Effective April 20. <http://www.epa.gov/region04/waste/ots/ecolbul.htm>

**APPENDIX D**

**FIELD SOPs AND FIELD FORMS**





# Tetra Tech NUS, Inc.

**PROJECT:** \_\_\_\_\_ **LOCATION:** \_\_\_\_\_  
**JOB & CTO #:** \_\_\_\_\_ **MOBILIZATION DATE:** \_\_\_\_\_  
**PROJECT MANAGER:** \_\_\_\_\_ **RETURN DATE:** \_\_\_\_\_

<b>FIELD PROJECT DEMOBILIZATION CHECKLIST</b>	
<p style="text-align: center; margin: 0;"><b>TRAVEL</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Airline reservations</li> <li><input type="checkbox"/> Hotel reservations/BOQs</li> <li><input type="checkbox"/> Vehicle rental</li> <li><input type="checkbox"/> Itinerary</li> <li><input type="checkbox"/> Phone/pager number</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>DRILLING/DPT/SURVEY</b></p> <p><b>Subcontractor</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> POC phone #/address</li> <li><input type="checkbox"/> Drill Specification RFP</li> <li><input type="checkbox"/> Contact (time &amp; place to meet)</li> <li><input type="checkbox"/> Confirm subcontract w/ TtNUS Procurement</li> <li><input type="checkbox"/> Health and Safety documentation for all personnel on site</li> <li><input type="checkbox"/> Copy of Drillers license</li> <li><input type="checkbox"/> Well / boring permits</li> </ul> <p><b>Utilities (2 weeks lead time)</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Contact Site POC (Date: _____)</li> <li><input type="checkbox"/> Contact Local "Call Before You Dig"</li> <li><input type="checkbox"/> Utility Clearance Form</li> </ul> <p><b>Forms</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Boring logs / Test Pit logs</li> <li><input type="checkbox"/> Well construction / development forms</li> <li><input type="checkbox"/> Daily activity forms</li> <li><input type="checkbox"/> IDW inventory</li> <li><input type="checkbox"/> IDW drum labels</li> <li><input type="checkbox"/> Chemical Inventory</li> <li><input type="checkbox"/> MSDS's</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>EQUIPMENT MOBILIZATION</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Equipment Requisition form completed / equipment ordered</li> <li><input type="checkbox"/> 3rd Party rental / misc. equipment ordered</li> <li><input type="checkbox"/> Equipment calibration forms</li> <li><input type="checkbox"/> Span / calibration gas and regulator</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>SAMPLING</b></p> <p><b>Forms</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Sample log sheets</li> <li><input type="checkbox"/> Low-flow purge data sheets</li> <li><input type="checkbox"/> COC records</li> <li><input type="checkbox"/> COC seals</li> <li><input type="checkbox"/> Sample labels (from database group)</li> </ul> <p><b>Laboratory</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> POC address/phone#</li> <li><input type="checkbox"/> Order bottles / preservatives</li> <li><input type="checkbox"/> Shipping address, also check Sat. address</li> <li><input type="checkbox"/> Bottle &amp; preservation req'ts from lab</li> </ul>	<p style="text-align: center; margin: 0;"><b>MISCELLANEOUS</b></p> <p><b>Schedule</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Plan field operations w/ Project manager</li> </ul> <p><b>Documents for Field Program</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Logbook(s)</li> <li><input type="checkbox"/> Field Sampling plan</li> <li><input type="checkbox"/> Health &amp; Safety plan</li> <li><input type="checkbox"/> Maps</li> <li><input type="checkbox"/> H &amp; S Guidance Manual</li> </ul> <p><b>Authorization</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Kick-off meeting held</li> <li><input type="checkbox"/> Gov't rate letter</li> <li><input type="checkbox"/> H&amp;S/OSHA 40-hour certificate</li> <li><input type="checkbox"/> 8-Hour Refresher Training Certificate</li> <li><input type="checkbox"/> Medical Clearance Letter</li> <li><input type="checkbox"/> Supervisory Training Certificate</li> <li><input type="checkbox"/> Health &amp; Safety Clearance Letter</li> <li><input type="checkbox"/> Full-size OSHA Poster</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>HYDROGEOLOGY EQUIPMENT</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Slug test/pumping test forms</li> <li><input type="checkbox"/> Groundwater elevation data sheets</li> <li><input type="checkbox"/> Graph paper</li> <li><input type="checkbox"/> Data Logger/transducer/data cable</li> <li><input type="checkbox"/> Existing well construction &amp; water level data</li> <li><input type="checkbox"/> M-Scope, slug</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>SHIPPING</b></p> <p><b>Forms</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> FedEx Airbills, local dropoff location &amp; hours</li> <li><input type="checkbox"/> FedEx Gov. Acct# (1771-8058-0)</li> <li><input type="checkbox"/> Lab Shipping Labels</li> <li><input type="checkbox"/> Warehouse Shipping Labels</li> <li><input type="checkbox"/> Blank Labels</li> </ul> <p><b>Supplies</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Tape</li> <li><input type="checkbox"/> Packing materials</li> <li><input type="checkbox"/> Baggies, Large garbage bags</li> </ul> <hr/> <p style="text-align: center; margin: 0;"><b>OTHER</b></p> <ul style="list-style-type: none"> <li><input type="checkbox"/> Site POC name/phone #</li> <li><input type="checkbox"/> Personnel information to POC</li> <li><input type="checkbox"/> Mobilization schedule to POC</li> <li><input type="checkbox"/> Site access authorizations</li> <li><input type="checkbox"/> Field office / trailer arrangements made</li> <li><input type="checkbox"/> Electric, phone hookups arranged</li> <li><input type="checkbox"/> Steel-toed boots, safety glasses, &amp; hard hat</li> <li><input type="checkbox"/> First aid equipment</li> <li><input type="checkbox"/> Insect repellent</li> </ul>

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.





**TETRA TECH NUS  
FIELD TASK MODIFICATION REQUEST FORM**

Project/Installation Name \_\_\_\_\_ CTO & Project Number \_\_\_\_\_ Task Mod. Number \_\_\_\_\_

Modification To (e.g. Work Plan) \_\_\_\_\_ Site/Sample Location \_\_\_\_\_ Date \_\_\_\_\_

Activity Description: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Reason for Change: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Recommended Disposition: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Field Operations Leader (Signature) \_\_\_\_\_ Date \_\_\_\_\_

Approved Disposition: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Project/Task Order Manager (Signature) \_\_\_\_\_ Date \_\_\_\_\_

Distribution:

Program/Project File – \_\_\_\_\_  
Project/Task Order Manager – \_\_\_\_\_  
Field Operations Leader – \_\_\_\_\_  
Other: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_







# Tetra Tech NUS, Inc.

PROJECT: \_\_\_\_\_ LOCATION: \_\_\_\_\_  
 JOB & CTO #: \_\_\_\_\_ MOBILIZATION DATE: \_\_\_\_\_  
 PROJECT MANAGER: \_\_\_\_\_ RETURN DATE: \_\_\_\_\_

<b>FIELD PROJECT PRE-MOBILIZATION CHECKLIST</b>	
<p style="text-align: center;"><b>TRAVEL</b></p> <p>___ Airline reservations            ___ Hotel reservations/BOQs            ___ Vehicle rental            ___ Itinerary            ___ Phone/pager number</p> <p style="text-align: center;"><b>DRILLING/DPT/SURVEY</b></p> <p><b>Subcontractor</b>            ___ POC phone #/address            ___ Drill Specification RFP            ___ Contact (time &amp; place to meet)            ___ Confirm subcontract w/ TtNUS Procurement            ___ Health and Safety documentation for all personnel on site            ___ Copy of Drillers license            ___ Well / boring permits</p> <p><b>Utilities (2 weeks lead time)</b>            ___ Contact Site POC (Date: _____)            ___ Contact Local "Call Before You Dig"            ___ Utility Clearance Form</p> <p><b>Forms</b>            ___ Boring logs / Test Pit logs            ___ Well construction / development forms            ___ Daily activity forms            ___ IDW inventory            ___ IDW drum labels            ___ Chemical Inventory            ___ MSDS's</p> <p style="text-align: center;"><b>EQUIPMENT MOBILIZATION</b></p> <p>___ Equipment Requisition form completed / equipment ordered            ___ 3rd Party rental / misc. equipment ordered            ___ Equipment calibration forms            ___ Span / calibration gas and regulator</p> <p style="text-align: center;"><b>SAMPLING</b></p> <p><b>Forms</b>            ___ Sample log sheets            ___ Low-flow purge data sheets            ___ COC records            ___ COC seals            ___ Sample labels (from database group)</p> <p><b>Laboratory</b>            ___ POC address/phone#            ___ Order bottles / preservatives            ___ Shipping address, also check Sat. address            ___ Bottle &amp; preservation req'ts from lab            ___ _____</p>	<p style="text-align: center;"><b>MISCELLANEOUS</b></p> <p><b>Schedule</b>            ___ Plan field operations w/ Project manager</p> <p><b>Documents for Field Program</b>            ___ Logbook(s)            ___ Field Sampling plan            ___ Health &amp; Safety plan            ___ Maps            ___ H &amp; S Guidance Manual</p> <p><b>Authorization</b>            ___ Kick-off meeting held            ___ Gov't rate letter            ___ H&amp;S/OSHA 40-hour certificate            ___ 8-Hour Refresher Training Certificate            ___ Medical Clearance Letter            ___ Supervisory Training Certificate            ___ Health &amp; Safety Clearance Letter            ___ Full-size OSHA Poster</p> <p style="text-align: center;"><b>HYDROGEOLOGY EQUIPMENT</b></p> <p>___ Slug test/pumping test forms            ___ Groundwater elevation data sheets            ___ Graph paper            ___ Data Logger/transducer/data cable            ___ Existing well construction &amp; water level data            ___ M-Scope, slug</p> <p style="text-align: center;"><b>SHIPPING</b></p> <p><b>Forms</b>            ___ FedEx Airbills, local dropoff location &amp; hours            ___ FedEx Gov. Acct# (1771-8058-0)            ___ Lab Shipping Labels            ___ Warehouse Shipping Labels            ___ Blank Labels</p> <p><b>Supplies</b>            ___ Tape            ___ Packing materials            ___ Baggies, Large garbage bags</p> <p style="text-align: center;"><b>OTHER</b></p> <p>___ Site POC name/phone #            ___ Personnel information to POC            ___ Mobilization schedule to POC            ___ Site access authorizations            ___ Field office / trailer arrangements made            ___ Electric, phone hookups arranged            ___ Steel-toed boots, safety glasses, &amp; hard hat            ___ First aid equipment            ___ Insect repellent            ___ _____            ___ _____</p>

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.



Project Site Name: \_\_\_\_\_ Sample ID No.: \_\_\_\_\_  
 Project No.: \_\_\_\_\_ Sample Location: \_\_\_\_\_  
 Sampled By: \_\_\_\_\_  
 Surface Soil C.O.C. No.: \_\_\_\_\_  
 Subsurface Soil  
 Sediment  
 Other: \_\_\_\_\_ Type of Sample:  
 QA Sample Type: \_\_\_\_\_  Low Concentration  
 High Concentration

**GRAB SAMPLE DATA:**

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

**COMPOSITE SAMPLE DATA:**

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

**SAMPLE COLLECTION INFORMATION:**

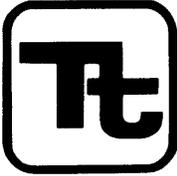
Analysis	Container Requirements	Collected	Other

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

MS/MSD Duplicate ID No.: \_\_\_\_\_ Signature(s): \_\_\_\_\_



TETRA TECH NUS,  
INC.

# STANDARD OPERATING PROCEDURES

Number	CT-04	Page	1 of 7
Effective Date	03/09/09	Revision	2
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston	<i>T.E. Johnston</i>	

Subject  
SAMPLE NOMENCLATURE

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

## 2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

**Program Manager** - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

**Project Manager** - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

**Field Operations Leader (FOL)** - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

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## 5.0 PROCEDURES

### 5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alphanumeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

<b>A or N 3 or 4 Characters</b>	<b>AAA 2 or 3 Characters</b>	<b>A or N 3 to 6 Characters</b>
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

- (1) Soil and sediment sample ID

<b>A or N 3 or 4 Characters</b>	<b>AAA 2 or 3 Characters</b>	<b>A or N 3 to 6 Characters</b>	<b>NNNN 4 Characters</b>
Site identifier	Sample type	Sample location	Sample depth

- (2) Aqueous (groundwater or surface water) sample ID

<b>A or N 3 or 4 Characters</b>	<b>AAA 2 or 3 Characters</b>	<b>A or N 3 to 6 Characters</b>	<b>NN 2 Characters</b>	<b>-A 1 Character</b>
Site identifier	Sample type	Sample location	Round number	Filtered sample only

- (3) Biota sample ID

<b>A or N 3 or 4 Characters</b>	<b>AAA 2 or 3 Characters</b>	<b>A or N 3 to 6 Characters</b>	<b>AA 2 Characters</b>	<b>NNN 3 Characters</b>
Site identifier	Sample type	Sample location	Species identifier	Sample group number

### 5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

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The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

### **5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS**

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

- A01 - Area of Concern (AOC) 1
- 125 - SWMU 125
- 000 - Base- or facility-wide sample (e.g., upgradient well)
- BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

- AH - Ash Sample

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- AS - Air Sample
- BM - Building Material Sample
- BSB - Biota Sample Full Body
- BSF - Biota Sample Fillet
- CP - Composite Sample
- CS - Chip Sample
- DS - Drum Sample
- DU - Dust Sample
- FP - Free Product
- IDW - Investigation-Derived Waste Sample
- LT - Leachate Sample
- MW - Monitoring Well Groundwater Sample
- OF - Outfall Sample
- RW - Residential Well Sample
- SB - Soil Boring Sample
- SD - Sediment Sample
- SC - Scrape Sample
- SG - Soil Gas Sample
- SL - Sludge Sample
- SP - Seep Sample
- SS - Surface Soil Sample
- ST - Storm Sewer Water Sample
- SW - Surface Water Sample
- TP - Test Pit Sample
- TW - Temporary Well Sample
- WC - Well Construction Material Sample
- WP - Wipe Sample
- WS - Waste/Solid Sample
- WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

- 001 - Monitoring well 1
- N32E92 - Grid location 32 North and 92 East
- D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

- BC - Blue Crab
- GB - Blue Gill
- CO - Corn
- SB - Soybean

#### 5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

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A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

## 5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

<b>AA</b>	<b>NNNNNN</b>	<b>NN</b>	<b>-F</b>
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank  
 RB = Rinsate Blank (Equipment Blank)  
 FD = Field Duplicate  
 AB = Ambient Conditions Blank  
 WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

## 5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

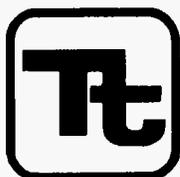
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The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

## **6.0 DEVIATIONS**

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.



TETRA TECH NUS, INC.

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Applicability Tetra Tech NUS, Inc.	
Prepared Management Information Systems Department	
Approved D. Senovich <i>[Signature]</i>	

Subject  
DATABASE RECORDS AND QUALITY ASSURANCE

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## 1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

## 2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

## 3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

## 4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

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**Earth Sciences Department Manager** - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

**FOL** - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

**Management Information Systems (MIS) Manager** - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request Form included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

**Program/Department Managers** - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

**Project Manager** - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

**Risk Assessment Department Manager** - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

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**Quality Assurance Reviewers** - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

**Quality Manager** - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

## 5.0 PROCEDURES

### 5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

### 5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

### 5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

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#### 5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

#### 5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of-Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

#### 5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

#### 5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

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editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

## 6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File  
PROJECT NUMBER: \_\_\_\_\_  
SITE NAME: \_\_\_\_\_  
DATE FILED: \_\_/\_\_/\_\_  
SUMMARY OF CONTENTS ENCLOSED  
BOX \_ OF \_

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

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**ATTACHMENT A**



**MIS REQUEST FORM**

Tetra Tech NUS, Inc.

Project Name:	Request Date:
CTO:	Date Data Available for Production:
Project Manager:	Request in Support of:
Requestor:	Database Lead:
Program/Client:	GIS Lead:
State/EPA Region:	Statistics Lead:
	Risk Lead:
Site Name(s) (Area, OU, etc.):	
Sampling Date(s):	
Matrix: <input type="checkbox"/> GW <input type="checkbox"/> SO <input type="checkbox"/> SD <input type="checkbox"/> SW <input type="checkbox"/> Other:	
<b>Labels:</b>	<input type="checkbox"/> Labels needed for an upcoming sampling event _____ Total # of Samples
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	
FOL _____	
<b>Data Entry:</b>	
<input type="checkbox"/> Chemical data needs to be entered from hardcopy _____ Estimated # of Samples	
<input type="checkbox"/> Chemical data needs to be formatted electronically _____	
<input type="checkbox"/> Field analytical data needs to be entered from hardcopy _____	
<input type="checkbox"/> Geologic data needs to be entered from hardcopy _____	
<input checked="" type="checkbox"/> Hydrology data needs to be entered from hardcopy _____	
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	
<b>Tables:</b>	
<input type="checkbox"/> Full Data Printout _____	
<input type="checkbox"/> Summary of Positive Hits _____	
<input type="checkbox"/> Occurance and Distribution _____ <input type="checkbox"/> with criteria	
<input type="checkbox"/> Sampling Analytical Summary: _____	
<input type="checkbox"/> Other: _____	
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	
<b>GIS:</b>	
<input type="checkbox"/> General Facility Location _____	
<input type="checkbox"/> Site Location _____	
<input type="checkbox"/> Potentiometric Contours/Groundwater Flow _____	
<input type="checkbox"/> Sample Location Proposed _____	
<input type="checkbox"/> Sample Location Existing _____	
<input type="checkbox"/> Tag Map Single Round _____	
<input type="checkbox"/> Tag Map Multiple Round _____	
<input type="checkbox"/> Isoconcentrations _____	
<input checked="" type="checkbox"/> Chart Map _____	
<input type="checkbox"/> 3D Visualization _____	
<input type="checkbox"/> EGIS CD _____	
<input type="checkbox"/> Other: _____	
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	
<b>Statistics:</b>	
<input type="checkbox"/> Yes _____	
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	
<b>Geostatistics:</b>	
<input type="checkbox"/> Yes _____	
Estimated Hours _____	Additional Instructions: _____
Due Date _____	
Complete ETS Charge No. _____	



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Applicability Tetra Tech NUS, Inc.	
Prepared Chemistry and Toxicology Department	
Approved T. Johnston 	

Subject  
DATA VALIDATION - CLP ORGANICS FOR SOLID  
AND AQUEOUS MATRICES

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### APPENDIX

#### **A SAMPLE CALCULATIONS**

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## 1.0 PURPOSE

This SOPC governs the validation of data generated by the following methods:

- Gas Chromatography/Mass Spectrometry
  - Volatile Organic Compounds (VOCs) by USEPA CLP Statement of Work (SOW) OLM04.3/OLC03.2/SOM01.1
  - Semivolatile Organic Compounds (SVOCS) by (USEPA CLP Statement of Work (SOW) (OLM04.3/OLC03.2/SOM01.1)
- Gas Chromatography
  - Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by USEPA CLP Statement of Work (SOW) OLM04.3/OLC03.2/SOM01.1)

## 2.0 APPLICABILITY

The applicability of each set of validation criteria is described in the appropriate section below.

## 3.0 PERSONNEL QUALIFICATIONS

The minimum qualifications of persons implementing this SOP are as follow:

- Education – Minimum of a bachelor's degree in chemistry or related physical/life science.
- Experience requirements include either operational experience with the analytical method or method data review training conducted under the direction of an experienced reviewer and performed on the subject matter data package. A record of the training will not be documented and kept on file but the data validation report produced under training will serve as the record.

## 4.0 CLP ORGANICS BY GC/MS

### 4.1 Volatiles (USEPA CLP Statement of Work (SOW) OLM04.3/OLC03.2/SOM01.1)

#### 4.1.1 Applicability

CLP volatile methodology is used to determine organic compounds in most matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, pastes, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

The CLP volatile Target Compound List (TCL) consists of the following compounds:

Acetone	1,4-Dichlorobenzene	Methyl Acetate
Benzene	1,2-Dibromo-3-Chloropropane	Methylcyclohexane
Bromodichloromethane	Dibromochloromethane	Methylene Chloride
Bromoform	Dichlorodifluoromethane	4-Methyl-2-Pentanone
Bromomethane	1,2-Dibromoethane	Methyl-t-butyl ether (MTBE)

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2-Butanone	1,1-Dichloroethane	Styrene
Carbon Disulfide	1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
Carbon Tetrachloride	1,1-Dichloroethene	Tetrachloroethene
Chlorobenzene	1,2-Dichloroethene (total)	Toluene
Chlorobromomethane	1,2-Dichloropropane	Trichlorofluoromethane
Chloroethane	cis-1,2-Dichloroethene	1,1,2-Trichloro-1,2,2-Trifluoroethane
Chloroform	cis-1,3-Dichloropropene	1,2,3-Trichlorobenzene
Chloromethane	trans-1,2-Dichloroethene	1,2,4-Trichlorobenzene
Cyclohexane	trans-1,3-Dichloropropene	1,1,1-Trichloroethane
1,2-Dibromoethane (EDB)	Ethyl Benzene	1,1,2-Trichloroethane
1,2-Dichlorobenzene	2-Hexanone	Trichloroethene
1,3-Dichlorobenzene	Isopropylbenzene	Vinyl Chloride
		Xylenes (total)

This method is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared according to the SOW.

#### 4.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

#### 4.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

#### 4.1.4 Sample Preparation

A purge-and-trap procedure is performed to prepare and extract volatile compounds from samples and to introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

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#### 4.1.5 Data Overview Prior to Validation

Before commencing validation, preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate numbers of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

NOTE: Unless specifically directed by client protocol, never annotate the laboratory data package.

- c. Prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses), and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

#### 4.1.6 Technical Evaluation Summary

Conduct all data evaluations in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. Reference the applicable documents during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

Evaluate general parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification concurrently with the parameters discussed in the following subsections.

##### 4.1.6.1 Holding Times and Sample Preservation Criteria

Verify that holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Calculate holding times from date of collection to date of analysis. Verify that samples are stored according to method requirements. Use the following rules:

- a. For unpreserved aqueous samples, apply a 7-day maximum holding time allowance for aromatic compounds, and with a 14-day maximum holding time allowance for chlorinated hydrocarbons.
- b. For aqueous samples preserved with hydrochloric acid (HCl) to pH 2 or below, apply a 14-day maximum holding time as the technical maximum holding time allowance .
- c. For soil samples in proper containers, apply a 14-day maximum holding time allowance.
- d. Verify that all samples were stored at 4°C ± 2 °C.

##### 4.1.6.2 Holding Time and Sample Preservation Action

- a. If maximum holding times are exceeded, qualify positive results in affected samples as estimated (J); and qualify nondetects as not detected/estimated (UJ). These results are usually assumed to be biased low unless prolonged storage causes a concentration increase, e.g., for degradation products which are also target analytes.
- b. If holding times are exceeded by a factor of more than two times the maximum holding time, qualify positive results as estimated (J); and qualify nondetects as rejected (UR). These exceedances are considered to be gross holding time exceedances.

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- c. If EPA Regional requirements apply, as in EPA Region III, apply the appropriate bias qualifiers as required; for example, detections and nondetects as biased low (L) or (UL), respectively.
- d. If samples are received above the required temperature, use professional judgment in applying qualifiers. Consider the length of time in storage, the inferred holding temperature, and other factors that could affect the target analyte concentrations.

#### 4.1.6.3 GC/MS Tuning Criteria

An analysis of an instrument performance check standard of Bromofluorobenzene must be performed at the beginning of each 12-hour period in which samples and standards are being analyzed.

- a. Verify that all ion abundance criteria below are within acceptance ranges on Form V or equivalent summary form:

m/z	Ion abundance criteria
50	8.0 – 40.0% of m/z 95
75	30.0 – 66.0% of m/z 95
95	Base peak, 100%
96	5.0 – 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 – 120.0% of m/z 95
175	5.0 – 9.0% of m/z 174
176	93.0 – 101.0% of m/z 174
177	5.0 – 9.0% of m/z 176

- b. Verify that all samples and standards were analyzed within the 12-hour period.

#### 4.1.6.4 GC/MS Tuning Action

- a. If mass assignment is in error, then reject all associated data (R) or (UR).
- b. If ion abundance criteria are not met, professional judgment may be used to determine the extent of data usability and whether qualifications are needed. The most critical abundances are m/z 95/96, 174/175, 174/176, and 176/177.
- c. If samples were analyzed beyond the 12-hour period, then qualify positive and nondetected results as estimated, (J) and (UJ) respectively.
- d. If the reviewer suspects that improper background subtraction techniques were used to generate a compliant tune, contact the laboratory and ask them to provide supporting evidence of tuning data. If the evidence is suitable, no further action is required. If proper evidence cannot be provided to support the tuning data, then professional judgment should be utilized to determine the usability of the associated data.

#### 4.1.6.5 Calibration Criteria

Verify the following:

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- a. Verify that an initial calibration was performed for each instrument used for analysis and for each type of medium and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.
- b. Verify that a continuing calibration was performed for each instrument used for analysis, for each type of medium, and that the continuing calibration was performed following the instrument tune.
- c. Review initial calibration Form VIs and the associated laboratory raw data to determine which compounds have:
  - 1) Average Relative Response Factors (RRFs) <0.050
  - 2) Percent Relative Standard Deviations (%RSDs) >30%.
- d. Circle noncompliant RRFs and %RSDs on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.
- e. Determine which samples are affected by non-compliant RRFs or %RSDs by reviewing the continuing calibration Form VIIs. Check the instrument identification and the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations.
- f. Review the sample listings given on the data package Form Vs to match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers.
- g. Review the continuing calibration Form VIIs and the associated laboratory raw data to determine which compounds have:
  - 1) RRFs <0.050
  - 2) Percent Differences (%Ds) >25%
- h. Circle the noncompliant RRFs and %Ds on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.
- i. For samples analyzed by OLC03.2, review the initial calibration and verify that the RRFs are greater than or equal to 0.010 for the compounds in Table 1 and are >0.050 for all other compounds:
- j. Review the initial calibration associated with OLC03.2 and verify that the %RSD is < 50% for the compounds in Table 1 and <30% for all other compounds:

**Table 1  
Volatile Compounds Exhibiting Poor Response**

Acetone	1,2-Dichloropropane
2-Butanone	1,2-Dibromo-3-chloropropane
Carbon Disulfide	4-Methyl-2-pentanone
Chloroethane	2-Hexanone
Chloromethane	Cyclohexane

- k. Review the continuing calibration associated with OLC03.2 and verify that the %Ds are < 50% for the compounds in Table 1 and <30% all other compounds.

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#### 4.1.6.6 Calibration Actions

- a. If any RRFs are <0.050, qualify all affected positive as estimated (J); qualify nondetects as nondetected rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- b. If any %RSD exceeds 30%, qualify affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate nondetects if the %RSD is >50% or reject nondetects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- c. If any %D exceeds 25%, qualify affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate nondetects if the %D is >50% or reject nondetects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- d. If any OLC03.2 compounds in Table 1 contain RRFs are <0.010 and if any other compounds are <0.050, qualify affected positive results as estimated (J); qualify nondetects as nondetected rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- e. If any OLC03.2 compounds in Table 1 are > 50% RSD and >30% for all other compounds, qualify affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ).
- f. If any OLC03.2 compounds in Table 1 are >50% D and >30% for all other compounds, qualify affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ).

#### 4.1.6.7 Blank Contamination Criteria

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed. Verify the following:

- a. A method or laboratory preparation blank must be analyzed during each 12-hour period.

The method blank should be free of contamination.

#### 4.1.6.8 Blank Contamination Action

- a. If a target compound is detected in any method blank:
  - 1) Select the maximum concentration of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!)
  - 2) Establish action levels for qualification (10X or 5X the maximum contaminant concentration depending upon whether or not the contaminant is a common contaminant). Common laboratory contaminants include methylene chloride, acetone, 2-butanone, and cyclohexane.

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3) Raise positive results that are less than the established blank action level to the Contract Required Quantitation Limit (CRQL) and qualify them as nondetect (U). In accordance with some USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results. In this case, qualify the results at the concentration detected instead of the CRQL.

- b. If a target compound was detected in a field quality control blank, carefully evaluate the associated samples to determine the appropriate action. Typically, field quality control blanks are not used to establish blank action levels but professional judgment may be used. When the reviewer decides to use a field quality control blank to qualify associated environmental samples, the guideline above must be followed.

#### 4.1.6.9 Surrogates Criteria

- a. Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.
- b. Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

#### 4.1.6.10 Surrogate Action

- a. If one or more surrogate recoveries exceed the upper quality control limit, qualify positive results as estimated (J); do not qualify Nondetects. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (K) qualifier may be used instead of (J) when qualifying positive results
- b. If one or more surrogate recoveries are below the lower quality control limit but are >10%, qualify positive and nondetected results as estimated (J) or nondetected estimated (UJ), respectively. These results are biased low. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (L, UL) qualifiers may be used instead of (J, UJ) when qualifying results
- c. If any surrogate recovery is <10%, qualify positive results as estimated (J); qualify nondetects as rejected (UR). These results are biased very low. The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- d. For OLC03.2 analyses, if a recovery is greater than the upper control limit, qualify positive results associated with that surrogate as estimated (J); do not qualify nondetects.
- e. For OLC03.2 analyses, if a recovery is greater than 20% but less than the lower quality control limit, qualify positive and nondetected results associated with that surrogate as estimated (J) or nondetected estimated (UJ), respectively.
- f. For OLC03.2 analyses, if a recovery is <20%, qualify positive results associated with that surrogate as estimated (J) and qualify nondetects as nondetected rejected (UR).

#### 4.1.6.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

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4.1.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. No action is generally taken on MS/MSD noncompliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results as estimated (J) and qualify nondetects as nondetected rejected (UR) in the unspiked sample.

4.1.6.13 Internal Standard Criteria

Evaluate internal standards by reviewing the data package Form VIIIs and the laboratory raw data. Verify the following:

- a. Internal standard areas fall within -50% or +100% for a given internal standard.
- b. For OLC03.2, internal standard areas fall within  $\pm 40\%$  a given internal standard.
- c. Retention times do not vary by more than  $\pm 30$  seconds.
- d. For OLC03.2, retention times do not vary by more than  $\pm 20$  seconds.

4.1.6.14 Internal Standard Action

- a. If the area count is > +100%, qualify positive results associated with a given internal standard as estimated (J); do not qualify nondetects.
- b. If the area count is < -50%, qualify positive and nondetected results associated with a given internal standard as estimated (J) or nondetected estimated (UJ), respectively.
- c. For OLC)3.2, if the area count is > +40%, qualify positive results associated with a given internal standard as estimated (J); do not qualify nondetects.
- d. For OLC03.2, if the area count is < -40%, qualify positive and nondetected results associated with a given internal standard as estimated (J) or nondetected estimated (UJ), respectively.
- e. If the retention time varies by more than  $\pm 30$  seconds carefully evaluate results, especially nondetected results. If deviations are severe, qualify the associated results as nondetected rejected (UR).
- f. For OLC03.2, if the retention time varies by more than  $\pm 20$  seconds carefully evaluate results, especially nondetected results. If deviations are severe, qualify the associated results as nondetected rejected (UR).

4.1.6.15 Tentatively Identified Compounds (TICs) Criteria

Verify that the laboratory reported TICs in the laboratory data package Form I VOA-TIC reports and the laboratory raw data.

4.1.6.16 Field Duplicate Precision Criteria

- a. Check samples to determine if field duplicates were included in the data package.

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- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50% for sample results greater than the reporting limit.

4.1.6.17 Field Duplicate Precision Action

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Qualify positive results showing imprecision as estimated (J) Bias for these results cannot be determined.
- b. If one result is positive and the other is nondetected and the positive result is greater than 2 times the reporting limit, qualify positive and nondetected results as estimated (J) or nondetected estimated (UJ), respectively.

4.1.6.18 Sample Result Verification Criteria

- a. Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

4.1.6.19 Sample Result Verification Action

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

4.1.6.20 Percent Solids Criteria

- a. Check the percent solids for each sample to identify any samples that contain <30% solids.

4.1.6.21 Percent Solids Action

- a. If any sample contains <30% solids, qualify positive and nondetected results as estimated (J) or nondetected estimated (UJ), respectively, due to the high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); qualify nondetected results as rejected (UR).

**4.1.7 Deliverables Guidance**

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for Data Validation

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Quality Assurance Officer (DV/QAO) review.

## 4.2 Semivolatiles (USEPA CLP Statement of Work (SOW) (OLM04.3/OLC03.2/SOM01.1)

### 4.2.1 **Applicability**

CLP semivolatile methodology is applicable to nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, pastes, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

The semivolatile TCL includes the following compounds:

Acenaphthene	2,2'-oxybis(1-Chloropropane)	Hexachlorocyclopentadiene
Acenaphthylene	Chrysene	Hexachloroethane
Acetophenone	Dibenzo(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Anthracene	Dibenzofuran	Isophorone
Atrazine	3,3'-Dichlorobenzidine	2-Methylnaphthalene
Benzaldehyde	2,4-Dichlorophenol	2-Methylphenol
Benzo(a)anthracene	Diethylphthalate	4-Methylphenol
Benzo(b)fluoranthene	2,4-Dimethylphenol	Naphthalene
Benzo(k)fluoranthene	Dimethylphthalate	2-Nitroaniline
Benzo(g,h,i)perylene	Di-n-butylphthalate	3-Nitroaniline
Benzo(a)pyrene	4,6-Dinitro-2-methylphenol	4-Nitroaniline
1,1'-Biphenyl	2,4-Dinitrophenol	Nitrobenzene
4-Bromophenyl-phenylether	2,4-Dinitrotoluene	2-Nitrophenol
Butylbenzylphthalate	2,6-Dinitrotoluene	4-Nitrophenol
Caprolactum	Di-n-octylphthalate	N-Nitroso-di-n-propylamine
Carbazole	bis(2-Ethylhexyl)phthalate	N-Nitroso-diphenylamine
4-Chloroaniline	Fluoranthene	Pentachlorophenol
bis(2-Chloroethoxy)methane	Fluorene	Phenanthrene
bis(2-Chloroethyl)ether	Hexachlorobenzene	Phenol
4-Chloro-3-methylphenol	Hexachlorobutadiene	Pyrene
2-Chloronaphthalene		1,2,4,5-Tetrachlorobenzene
2-Chlorophenol		2,4,5-Trichlorophenol
4-Chlorophenyl-phenylether		2,4,6-Trichlorophenol

The preceding method is based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures.

### 4.2.2 **Interferences**

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts that cause elevated baselines and lead to potential misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source depending upon the diversity of the industrial complex or waste being sampled.

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#### 4.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted once per 20 samples of a similar matrix to determine the effects of sample matrix upon the compounds of interest.

#### 4.2.4 Sample Preparation

Prior to GC/MS analysis, aqueous samples are acidified to pH 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted with 1:1 methylene chloride/acetone using a sonication procedure. Cleanup by Gel Permeation Chromatography (GPC) is required for solid sample extracts.

#### 4.2.5 Data Overview to Validation

Before commencing validation, preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate numbers of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

NOTE: Unless specifically directed by client protocol, never annotate the laboratory data package.

- c. Prepare working copies of all Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

#### 4.2.6 Technical Evaluation Summary

Conduct all data evaluations in accordance with the appropriate USEPA Regional protocols (when applicable) and/or specified client contract requirements. Reference the applicable documents during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

Evaluate general parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification concurrently with the parameters discussed in the following subsections.

##### 4.2.6.1 Holding Times and Sample Preservation Criteria

Verify that holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Calculate holding times for extraction from date of collection to date of extraction. Verify that samples are stored to method requirements. Use the following rules:

- a. For aqueous samples, use a 7-day maximum holding time until extraction.
- b. For soil samples, use a 14-day maximum holding time until extraction.

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- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.
- d. Verify that all samples were stored at 4°C ± 2 °C prior to extraction.
- e. Verify that all extracts were stored at 4°C ± 2 °C.

#### 4.2.6.2 Holding Times and Sample Preservation Action

- a. If holding times are exceeded, qualify positive results in affected samples as estimated (J); qualify nondetects as nondetected estimated(UJ). These results are usually assumed to be biased low unless prolonged storage causes a concentration increase, e.g., for degradation products which are also target analytes.
- b. If holding times are exceeded by a factor of more than two times the required time, qualify positive results as estimated (J); qualify nondetects as nondetected rejected (UR). These exceedances are considered to be gross holding time exceedances.
- c. If EPA Regional requirements apply, as in EPA Region III, apply the appropriate bias qualifiers as required; for example, detections and as nondetects biased low (L) or (UL), respectively.
- d. If samples are received above the required temperature, use professional judgment in applying qualifiers. Consider the length of time in storage, the inferred holding temperature, and other factors that could affect the target analyte concentrations.

#### 4.2.6.3 GC/MS Tuning Criteria

An analysis of an instrument performance check standard of Decafluorotriphenylphosphine (DFTPP) must be performed at the beginning of each 12-hour period in which samples and standards are being analyzed.

- a. Verify that all ion abundance criteria below are within acceptance ranges on Form V or equivalent summary form:

m/z	Ion abundance criteria
51	30.0 – 80.0% of m/z 198
68	Less than 2.0% of m/z 198
69	Mass 69 relative abundance
70	Less than 2.0% of m/z 69
127	25.0 – 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base Peak 100%
199	5.0 – 9.0% of m/z 198
275	10.0 – 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 – 110.0% of m/z 198
443	15.0 – 24.0% of m/z 442

- b. Verify that all samples and standards were analyzed within the 12-hour period.

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#### 4.2.6.4 GC/MS Tuning Action

- a. If mass assignment is in error, then reject all associated data (R) or (UR).
- b. If ion abundance criteria are not met, professional judgment may be used to determine the extent of data usability and whether qualifications are needed. The most critical abundances are m/z 199/198 and 442/443.
- c. If the relative abundance of m/z 365 is low or is zero this is an indication of an unsuitable instrument zero. Detection limits may be affected and nondetected results should be qualified (UJ).
- d. If samples were analyzed beyond the 12-hour period, then qualify positive and nondetected results as estimated, (J) and (UJ) respectively.
- e. If the reviewer suspects that improper background subtraction techniques were used to generate a compliant tune, contact the laboratory and ask them to provide supporting evidence of tuning data. If the evidence is suitable, no further action is required. If proper evidence cannot be provided to support the tuning data, then professional judgment should be utilized to determine the usability of the associated data.

#### 4.2.6.5 Calibration Criteria

Verify the following:

- a. Verify that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.
- b. Verify that a continuing calibration was performed for each instrument used for analysis and that the continuing calibration was performed following the instrument tune.
- c. Review initial calibration Form VIs and the associated laboratory raw data to determine which compounds have:
  - 1) Average Relative Response Factors (RRFs) <0.050
  - 2) Percent Relative Standard Deviations (%RSDs) >30%.
- d. Circle these noncompliant RRFs and %RSDs on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.
- e. Determine which samples are affected by non-compliant RRFs or %RSDs by reviewing the continuing calibration Form VIIs. Check the instrument identification and the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers.
- f. Review the continuing calibration Form VIIs and the associated laboratory raw data to determine which compounds have:
  - 1) RRFs <0.050
  - 2) Percent Differences (%Ds) >25%

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- g. Circle the noncompliant RRFs and %Ds on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.
- h. For samples analyzed by OLC03.2, review the initial calibration and verify that the RRFs are greater than or equal to 0.010 for the compounds in Table 2 and are >0.050 for all other compounds:
- i. Review the initial calibration associated with OLC03.2 and verify that the %RSDs are < 50% for the compounds in Table 2, <30% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol, and <20.5% for all other compounds.

**Table 2**  
**Semivolatile Compounds Exhibiting Poor Response**

2,2'-oxybis(1-Chloropropane)	Benzaldehyde
4-Chloroaniline	Pentachlorophenol
Hexachlorobutadiene	4-Nitroaniline
Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol
2-Nitroaniline	N-Nitroso-diphenylamine
3-Nitroaniline	3,3'-Dichlorobenzidine
2,4-Dinitrophenol	4-Nitrophenol
Acetophenone	Caprolactum

- j. Review the continuing calibration associated with OLC03.2 and verify that the %Ds are ≤ 50% for the following compounds in Table 2, ≤30% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol, and ≤25.0% for all other compounds.

#### 4.2.6.6 Calibration Actions

- a. If any RRFs are <0.050, qualify all affected positive results as estimated (J); qualify nondetects as nondetected rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- b. If any %RSD exceeds 30%, qualify all affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate nondetects if the %RSD is >50% or reject nondetects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- c. If any %D exceeds 25%, qualify all affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate nondetects if the %D is >50% or reject nondetects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- d. If any OLC03.2 compounds in Table 2 contain RRFs are <0.010 and if any other compounds are <0.050 qualify affected positive results as estimated (J); qualify nondetects as rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- e. If any OLC03.2 compounds in Table 2 contain %RSDs that are > 50%, >30% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol, and >20.5% for all other compounds. qualify affected positive results as estimated (J); qualify nondetects as nondetected estimated (UJ).

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- f. If any OLC03.2 compounds in Table 2 contain %Ds that are >50% >30% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol, and >25.0% for all other compounds, qualify positive results as estimated (J); qualify nondetects as nondetected estimated (UJ).

#### 4.2.6.7 Blank Contamination Criteria

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed. Verify the following:

- a. A method or laboratory preparation blank must be analyzed during each 12-hour period.
- b. The method blank should be free of contamination.
- c. Note that unlike volatile fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatiles method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

#### 4.2.6.8 Blank Contamination Action

- a. If a target compound is detected in any method blank:
  - 1) Select the maximum concentration of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!)
  - 2) Establish action levels for qualification (10X or 5X the maximum contaminant concentration depending upon whether or not the contaminant is a common contaminant).. Common laboratory contaminants include phthalates. For method OLC03.2, the action level is 5 times the maximum contaminant concentration for phthalates.
  - 3) Raise positive results that are less than the established blank action level to the Contract Required Quantitation Limit (CRQL) and qualify them as nondetect (U). In accordance with some USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results. In this case, qualify results at the concentration detected instead of the CRQL.
- b. If a target compound was detected in a field quality control blank, carefully evaluate the associated samples to determine the appropriate action. Typically, field quality control blanks are not used to establish blank action levels but professional judgment may be used. When the reviewer decides to use a field quality control blank to qualify associated environmental samples, the guideline above must be followed.

#### 4.2.6.9 Surrogates Criteria

Semivolatiles compounds are divided into two fractions, base-neutral compounds and acid-extractable compounds. Each fraction of compounds has its own associated surrogates. Phenolic compounds are included in the acid fraction and all remaining compounds are included in the base-neutral fraction.

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Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.

Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

#### 4.2.6.10 Surrogate Action

- a. If two or more surrogate recoveries for a given fraction exceed the upper quality control limit, qualify positive results in that fraction as estimated (J); do not qualify nondetects. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (K) qualifier may be used instead of (J) when qualifying positive results
- b. If two or more surrogate recoveries for a given fraction are below the lower quality control limit but are >10%, qualify positive and nondetected results in the associated fraction as estimated (J) or nondetected estimated, respectively(UJ). These results are biased low. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (L,UL) qualifiers may be used instead of (J, UJ) when qualifying results
- c. If any surrogate recovery is <10% in a given fraction, qualify positive results in that fraction as estimated (J); qualify nondetects as nondetected rejected (UR). These results are biased very low. The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- d. For OLC03.2 analyses, if a surrogate recovery is greater than the upper control limit, qualify positive results associated with that surrogate as estimated (J); do not qualify nondetects qualified.
- e. For OLC03.2 analyses, if a surrogate recovery is less than the lower quality control limit, qualify positive and nondetected results associated with that surrogate as estimated (J) or nondetected estimated (UJ), respectively.
- f. For OLC03.2 analyses, if a recovery is <10%, qualify positive results associated with that surrogate as estimated (J) and qualify nondetects as rejected (UR).

#### 4.2.6.11 Matrix Spike/Matrix Spike Duplicate Criteria

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

#### 4.2.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. Take no action based on MS/MSD noncompliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results in the unspiked sample as estimated (J) and qualify nondetects as nondetected rejected (UR) sample.

#### 4.2.6.13 Internal Standard Criteria

Evaluate internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory

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raw data. Verify the following:

- a. Internal standard areas fall within -50% or +100% for a given internal standard.
- b. Retention times do not vary by more than  $\pm 30$  seconds.

#### 4.2.6.14 Internal Standard Action

- a. If the area count is  $> +100\%$ , qualify positive results associated with a given internal standard as estimated (J); do not qualify nondetects.
- b. If the area count is  $< -50\%$ , qualify positive and nondetected results associated with a given internal standard as estimated (J) or nondetected estimated (UJ), respectively.
- c. If the retention time varies by more than  $\pm 30$  seconds carefully evaluate results, especially nondetected results. If deviations are severe, qualify the associated results as nondetected rejected (UR).

#### 4.2.6.15 Tentatively Identified Compounds (TICs)

Verify that the laboratory reported TICs in the laboratory data package Form I SVOA-TIC reports and the laboratory raw data.

#### 4.2.6.16 Field Duplicate Precision Criteria

- a. Check samples to determine if field duplicates were included in the data package.
- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be  $< 30\%$ ; for soil matrix results,  $< 50\%$  for sample results greater than the reporting limit.

#### 4.2.6.17 Field Duplicate Precision Action

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Qualify positive results for compounds showing imprecision are qualified as estimated (J) Bias for these results cannot be determined.
- b. If one result is positive and the other is nondetected and the positive result is greater than 2 times the reporting limit, qualify positive and nondetected results as estimated (J) and or nondetected estimated (UJ), respectively.

#### 4.2.6.18 Sample Result Verification Criteria

- a. Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

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#### 4.2.6.19 Sample Result Verification Action

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

#### 4.2.6.20 Percent Solids Criteria

- a. Check the percent solids for each sample to identify any samples that contain <30% solids.

#### 4.2.6.21 Percent Solids Action

- a. If any sample contains <30% solids, qualify positive and nondetected results as estimated (J) or nondetected estimated (UJ), respectively, due to the high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); qualify nondetected results as rejected (UR).

### 4.2.7 **Deliverables Guidance**

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for Data Validation Quality Assurance Officer (DV/QAO) review.

## 5.0 **CLP ORGANICS BY GC**

### 5.1 **Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) (USEPA CLP Statement of Work (SOW) OLM04.3/OLC03.2/SOM01.1)**

#### 5.1.1 **Applicability**

CLP methodology is used to determine the concentration of certain organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices. Specifically, the CLP TCL includes the following substances:

Aldrin	Dieldrin	Methoxychlor
alpha-BHC	Endosulfan I	Toxaphene
Alpha chlordane	Endosulfan II	Aroclor-1016
beta-BHC	Endosulfan sulfate	Aroclor-1221
delta-BHC	Endrin	Aroclor-1232
gamma-BHC (Lindane)	Endrin aldehyde	Aroclor-1242
Gamma Chlordane	Endrin ketone	Aroclor-1248
4,4'-DDD	Heptachlor	Aroclor-1254
4,4'-DDE	Heptachlor epoxide	Aroclor-1260

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#### 4,4'-DDT

CLP methodology for organochlorine pesticides and PCBs is a Gas Chromatographic (GC) procedure in which samples are first extracted and then analyzed by direct injection. The compounds of interest are analyzed via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used).

#### 5.1.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts that cause elevated baselines and lead to potential misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the sample will vary considerably and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

#### 5.1.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicates should also be employed.

#### 5.1.4 Sample Preparation

Prior to GC analysis, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel or a continuous liquid-liquid extractor. Solid samples are extracted with hexane:acetone (1:1) using sonication procedures.

#### 5.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

NOTE: Unless specifically directed by client protocol, never annotate the laboratory data package.

- c. Prepare working copies of all Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

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### 5.1.6 Technical Evaluation Summary

Conduct all data evaluations in accordance with applicable USEPA Regional protocols (when applicable) and/or specific client contract requirements. Reference the applicable documents during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

Evaluate general parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification concurrently with the parameters discussed in the following subsections.

#### 5.1.6.1 Holding Times and Sample Preservation Criteria

Verify that holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Calculate holding times for extraction from date of collection to date of extraction. Verify that samples are stored to method requirements. Use the following rules:

- a. For aqueous samples, use a 7-day maximum holding time until extraction.
- b. For soil samples, use a 14-day maximum holding time until extraction.
- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.
- d. Verify that all samples were stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  prior to extraction.
- e. Verify that all extracts were stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

#### 5.1.6.2 Holding Times and Sample Preservation Action

- a. If holding times are exceeded, qualify positive results in affected samples as estimated (J); qualify nondetects as nondetected estimated (UJ). These results are usually assumed to be biased low unless prolonged storage causes a concentration increase, e.g., for degradation products which are also target analytes. .
- b. If holding times are exceeded by a factor of more than two times the required time, qualify positive results as estimated (J); qualify nondetects as nondetected rejected (UR). These exceedances are considered to be gross holding time exceedances.
- c. If EPA Regional requirements apply, as in EPA Region III, apply the appropriate bias qualifiers as required; for example, detections and nondetects biased low (L) or (UL), respectively.
- d. If samples are received above the required temperature, use professional judgment in applying qualifiers. Consider the length of time in storage, the inferred holding temperature, and other factors that could affect the target analyte concentrations..

#### 5.1.6.3 Instrument Performance Check Criteria

At the beginning of the initial calibration sequence, a Resolution Check Mixture is analyzed. A Performance Evaluation Mixture (PEM) is also analyzed at the beginning and end of the initial calibration sequence. After the initial calibration is established the PEM standard is analyzed at the beginning of every other 12-hour analytical period. During the review of the instrument performance check, verify the

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following:

- a. Check that the frequency of both the Resolution Check Mix and the PEM standard satisfy the analytical sequence criteria stated above.
- b. Check that resolution between any two adjacent peaks in the Resolution Check Mix are greater than or equal to 60% on each column.
- c. Check that resolution between any two adjacent peaks in the PEM are greater than or equal to 90% on each column
- d. Check that the breakdown of 4,4'-DDT and Endrin in each PEM is <20.0%
- e. Check that the combined breakdown of 4,4'-DDT and Endrin in each PEM is <30.0%

#### 5.1.6.4 Instrument Performance Check Action

- a. If the resolution criterion is not met in either the Resolution Check Mix or the PEM, qualify positive results as estimated (J). Use professional judgment to determine if nondetected data should be qualified as nondetected rejected (UR).
- b. If 4,4'-DDT breakdown exceeds 20%, qualify positive results for 4,4'-DDE and 4,4'-DDD as estimated (J).
- c. If 4,4'-DDT breakdown exceeded 20% and 4,4'-DDT was not detected but 4,4'-DDD and 4,4'-DDE were detected, then qualify positive results for 4,4'-DDD and 4,4'-DDE as presumptively present (NJ) and qualify the nondetected result for 4,4'-DDT nondetected rejected (UR).
- d. If Endrin breakdown exceeds 20%, qualify positive results for Endrin aldehyde and Endrin Ketone as estimated (J).
- e. If Endrin breakdown exceeded 20% and Endrin was not detected but Endrin aldehyde and Endrin Ketone were detected, then qualify positive results for Endrin aldehyde and Endrin Ketone as presumptively present (NJ) and qualify the nondetected result for Endrin as nondetected rejected (UR).
- f. If the combined breakdown of 4,4'-DDT and Endrin exceed 30.0%, qualify results as described above based upon the degree of individual breakdown.

#### 5.1.6.5 Calibration Criteria

Individual Mixes A and B are analyzed to establish an initial calibration curve on each GC and instrument used for analysis. The Individual Mixes A and B are analyzed at periodic intervals in the analytical sequence as calibration verification standards. During the review of the analytical calibration sequence, verify the following:

- a. Individual Mixes A and B were analyzed at a Low, Medium, and High level on each GC column and instrument.
- b. The %RSD for all single component pesticides are <20.0%
- c. Individual Mixes A and B were analyzed every other 12-hour period.

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- d. For method OLC03.2, the %RSD for delta BHC and alpha BHC are <25.0%
- e. The calibration verification %Ds for all single component pesticides are <25.0%.

5.1.6.6 Calibration Action

- a. If the %RSD of any compound in Individual Mix A or B exceeds 20%, qualify positive results as estimated (J) reported on the column which on noncompliance occurs. If the noncompliance occurs on both analytical columns, qualify nondetects as nondetected estimated (UJ).
- b. For OLC03.2, if the %RSD for delta BHC or alpha BHC exceeds 25.0%, qualify positive results as estimated (J) reported on the column which on noncompliance occurs. If the noncompliance occurs on both analytical columns qualify nondetects as nondetected estimated (UJ).
- c. If the %D of any compound in Individual Mix A or B exceeds 20%, qualify positive results as estimated (J) reported on the column which on noncompliance occurs. If the noncompliance occurs on both analytical columns qualify nondetects as nondetected estimated (UJ).

5.1.6.7 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed. Verify the following:

- a. A method or laboratory preparation blank must be analyzed during each 12-hour period.
- b. The method blank should be free of contamination.
- c. Note that unlike volatile fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one pesticide/PCB method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).
- d. Instrument blanks must be analyzed at the beginning and end of each 12-hour analytical sequence.

5.1.6.8 Blank Contamination Action

- a. If a target compound is detected in any preparation or instrument blank:
  - 1) Select the maximum concentration of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!)
  - 2) Establish an action level for qualification of 5X the maximum contaminant concentration.
  - 3) Raise positive results that are less than the established blank action level to the Contract Required Quantitation Limit (CRQL) and qualify them as nondetect (U). In accordance with some

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USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results. In this case, qualify results at the concentration detected instead of the CRQL.

- b. If a target compound was detected in a field quality control blank, carefully evaluate the associated samples to determine the appropriate action. Typically, field quality control blanks are not used to establish blank action levels but professional judgment may be used. When the reviewer decides to use a field quality control blank to qualify associated environmental samples, the guideline above must be followed.

#### 5.1.6.9 Surrogate Criteria

Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.

- a. Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.
- b. Verify that the decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII are within +/- 0.10 minutes for DCB and 0.05 minutes for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peaks.

#### 5.1.6.10 Surrogate Action

- a. If any surrogate recovery exceeds the upper quality control limit, qualify positive results in that fraction as estimated (J); do not qualify nondetects. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (K) qualifier may be used instead of (J) when qualifying positive results
- b. If any surrogate recovery is below the lower quality control limit but are >10%, qualify positive and nondetected results in the associated fraction as estimated (J) or nondetected estimated (UJ), respectively. These results are biased low. A bias qualifier may be used in certain Regions. In accordance with some USEPA Regional protocol, the (L,UJ) qualifiers may be used instead of (J, UJ) when qualifying results
- c. If any surrogate recovery is <10% in a given fraction, qualify positive results in that fraction as qualified as estimated (J); qualify nondetects as nondetected rejected (UR). These results are biased very low. The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- d. If any surrogate retention times have drifted outside of the specified retention time windows, use professional judgment to evaluate the potential impact and usability. Consider the degree of drift and any other factors that are relevant.

#### 5.1.6.11 Matrix Spike/Matrix Spike Duplicates

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

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5.1.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. Take no action based on MS/MSD noncompliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results in the unspiked sample as estimated (J) and qualify nondetects as nondetected rejected (UR).

5.1.6.13 Field Duplicate Precision Criteria

- a. Check samples to determine if field duplicates were included in the data package.
- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50% for sample results greater than the reporting limit.

5.1.6.14 Field Duplicate Precision Action

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J). Bias for these results cannot be determined.
- b. If one result is positive and the other is nondetected and the positive result is greater than 2 times the reporting limit, qualify positive and nondetected results as estimated (J) or (UJ), respectively.

5.1.6.15 Sample Result Verification Criteria

- a. Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

5.1.6.16 Sample Result Verification Action

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

5.1.6.17 Percent Solids Criteria

- a. Check the percent solids for each sample to identify any samples that contain <30% solids.

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#### 5.1.6.18 Percent Solids Action

- a. If any sample contains <30% solids, qualify positive and nondetected results as estimated (J) or nondetected estimated (UJ), respectively, due to the high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); qualify nondetected results as rejected (UR).

#### 5.1.6.19 Target Compound Identification Criteria

Verify the following:

- a. Check that the retention times of surrogates and target compounds fall within the retention time windows determined from the initial calibration.
- b. Check that reported target compounds were analyzed confirmed on two dissimilar columns.
- c. Check that the %D between positive results is < 25 % for single component compounds.
- d. Compare the chromatographic pattern of positively reported multicomponent compounds(e.g. Aroclors) to standards in order to verify pattern agreement and proper identification.
- e. Check that the lower of the two column positive results are reported.

#### 5.1.6.20 Target Compound Identification Action

- a. If the retention times of any compounds fall outside of the established retention time windows, use professional judgment to determine data usability.
- b. If the %D between columns for positive results exceeds 25% but is <100%, qualify the positive result as estimated (J).
- c. If the %D between columns for positive results is >100%, then the positive result may be rejected (R). However, professional judgment should be used to evaluate the chromatogram prior to rejecting the positive result. It should be noted that this action is limited to single component compounds.
- d. If the %D between columns for multicomponent compounds is >25% but <500%, qualify the positive result as estimated (J). If the %D is >500%, use professional judgment to determine if rejection (R) is necessary.

#### 5.1.7 **Deliverables Guidance**

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for Data Validation Quality Assurance Officer (DV/QAO) review.

#### 6.0 **REFERENCES**

Department of Defense (DoD) Environmental Data Quality Workgroup, 2006. Quality Systems Manual

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(QSM) for Environmental Laboratories, Final Version 3, January.

EPA540/R-99-008, U.S. EPA, 1999. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October.

EPA540/R-00-006, U.S. EPA, 2001. USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review, June.

U.S. EPA, 1999. USEPA Contract Laboratory Program Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM04.2, May.

U.S. EPA, 2000. USEPA Contract Laboratory Program Statement of Work for Organic Analysis of Low Concentration Media, OLC03.2, December.

U.S. EPA, 2005. USEPA Contract Laboratory Program Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, SOM01.1, May.

U.S. EPA Region I, 1996. Part II Volatile/Semivolatile Data Validation Functional Guidelines, December.

U.S. EPA Region I, 2004. Part III Pesticide/PCB Data Validation Functional Guidelines, February.

U.S. EPA Region III, 1994. Region III Modifications to the National Functional Guidelines for Organic Data Review, Multi-Media, Multi-Concentration, September.

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## APPENDIX A SAMPLE CALCULATIONS

Exhibit D Low/Medium Volatiles -- Section 11  
Data Analysis and Calculations (Con't)

11.2.1.2 Water

EQ. 7 Water Concentration Calculation

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{RRF}) (V_o)}$$

Where,

$A_x$  = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and DMCs are listed in Table 2.

$A_{is}$  = Area of the characteristic ion (EICP) for the internal standard. The target compounds are listed with their associated internal standards in Table 3.

$I_s$  = Amount of internal standard added, in ng.

$\overline{RRF}$  = Mean Relative Response Factor from the initial calibration.

$V_o$  = Total volume of water purged, in mL.

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e.,  $V_o$  above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5.0 mL with reagent water and purged,  $DF = 5.0 \text{ mL} / 2.0 \text{ mL} = 2.5$ . If no dilution is performed,  $DF = 1.0$ .

11.2.1.3 Low-Level Soil/Sediment

EQ. 8 Low-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } (\mu\text{g/Kg}) \text{ (dry weight basis)} = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{RRF}) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$ , and DF are as given for water, Equation 7.

$\overline{RRF}$  = Mean Relative Response Factor from the heated purge of the initial calibration.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

$W_s$  = Weight of sample added to the purge tube, in g.

11.2.1.4 Medium-Level Soil/Sediment

D-41/LOW-MED VOA

SOM01.1 (5/2005)

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Exhibit D Low/Medium Volatiles -- Section 11  
Data Analysis and Calculations (Con't)

EQ. 9 Medium-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } \mu\text{g/Kg (dry weight basis)} = \frac{(A_x) (I_s) (AV_t) (1000) (DF)}{(A_{is}) (\overline{RRF}) (V_a) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$  are as given for water, Equation 7.

$\overline{RRF}$  = Mean Relative Response Factor from the **ambient** temperature purge of the initial calibration.

$AV_t$  = Adjusted total volume of the methanol extract plus soil water in milliliters (mL) determined by:

$$AV_t = V_t + \{W_s - [W_s(D)]\}$$

Where  $V_t$  = total volume of methanol extract in milliliters (mL). This volume is typically 10 mL, even though only 1.0 mL is transferred to the vial in Section 10.1.5.5. The quantity derived from  $\{W_s - [W_s(D)]\}$  is the soil water volume and is expressed in mL.

$V_a$  = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100  $\mu\text{L}$ ), in microliters ( $\mu\text{L}$ ) added to reagent water for purging.

$W_s$  = Weight of soil/sediment extracted, in g.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

DF = Dilution Factor. The DF for analysis of soil/sediment samples for volatiles by the medium-level method is defined as:

$$\frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

11.2.1.5 For water, low-level and medium-level soil/sediment samples, xylenes are to be reported as "m,p-xylenes" and "o-xylene". Because m- and p-xylene isomers coelute, special attention must be given to the quantitation of the xylenes. In quantitating sample concentrations, be sure to use the correct corresponding Relative Response Factor (RRF) values.

NOTE: The area of each peak (i.e., the peaks for o-xylene and m,p-xylene) must appear on the complete quantitation report.

11.2.1.6 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene are to be reported separately.

11.2.1.7 Secondary ion quantitation is allowed **only** when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

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compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet Quality Control (QC) criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instance of manual integration must be documented in the SDG Narrative.

11.2.1.3 In all instances where the data system report has been edited or where manual integration or quantitation has been performed, the GC/MS Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "M" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and DMCs.

11.2.1.4 The requirements listed in Sections 11.2.1.1 - 11.2.1.3 apply to all standards, samples, and blanks.

11.2.1.5 The Mean Relative Response Factor ( $\overline{RRF}$ ) from the initial calibration is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reason in the SDG Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a  $\overline{RRF}$  is calculated using the secondary ion.

11.2.1.6 Calculate the concentration in the sample using the  $\overline{RRF}$  and Equations 5 and 6.

11.2.1.6.1 Water

EQ. 5 Concentration of Water Sample

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{RRF}) (V_o) (V_i)}$$

Where,

$A_x$  = Area of the characteristic ion for the compound to be measured.

$A_{is}$  = Area of the characteristic ion for the internal standard.

$I_s$  = Amount of internal standard injected in ng.

$V_o$  = Volume of water extracted in mL.

$V_i$  = Volume of extract injected in  $\mu\text{L}$ .

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$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$  (If GPC Cleanup is performed,  $V_t = V_{out}$ ).

$\overline{RRF}$  = Mean Relative Response Factor determined from the initial calibration standard.

$\text{GPC} = \frac{V_{in}}{V_{out}}$  = GPC factor. (If no GPC is performed,  $\text{GPC} = 1$ ).

$V_{in}$  = Volume of extract loaded onto GPC column.

$V_{out}$  = Volume of extract collected after GPC cleanup.

DF = Dilution Factor. The DF for analysis of water samples for semivolatiles by this method is defined as follows:

$$\text{DF} = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed,  $\text{DF} = 1.0$ .

11.2.1.6.2 Soil/Sediment

EQ. 6 Concentration of Soil/Sediment Sample

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{RRF}) (V_i) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$ ,  $V_{in}$ , and  $V_{out}$  are as given for water, above.

$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$   
(If no GPC Cleanup is performed, then  $V_t = 1000 \mu\text{L}$ .  
If GPC Cleanup is performed, then  $V_t = V_{out}$ ).

$V_i$  = Volume of the extract injected in  $\mu\text{L}$ .

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

$W_s$  = Weight of sample extracted in g.

$\text{GPC} = \frac{V_{in}}{V_{out}}$  = GPC Factor

$\overline{RRF}$  = Mean Relative Response Factor determined from the initial calibration standard.

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DF = Dilution Factor. The DF for analysis of soil/sediment samples for semivolatiles by this method is defined as follows:

$$DF = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

A GPC factor of 2.0 is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 0.5 mL maintains the sensitivity of the soil/sediment method.

11.2.2 Non-Target Compound

An estimated concentration for non-target compounds tentatively identified shall be quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used. The equations for calculating concentration are the same as Equations 5 and 6. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compounds to be measured and the internal standard. An RRF of 1 is to be assumed. The resulting concentration shall be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all TICs as well as those identified as unknowns.

11.2.3 CRQL Calculations

11.2.3.1 Water Samples

EQ. 7 Aqueous Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(V_x)(V_t)(DF)}{(V_o)(V_c)}$$

Where,

$V_t$ , DF, and  $V_o$  are as given in Equation 5.

$V_x$  = Contract sample volume (1000 mL).

$V_c$  = Contract concentrated extract volume (1000  $\mu$ L if GPC is not performed. If GPC was performed, then  $V_c = V_{out}$ ).

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- 11.2.1.4 The Contractor must quantitate Toxaphene based on the Mean Calibration Factors (CFs) from the most recent initial calibration.
- 11.2.1.5 The chromatograms of all samples [including Laboratory Control Samples (LCSs), Matrix Spikes and Matrix Spike Duplicates (MS/MSDs)], standards, and required blanks must be reviewed by a qualified pesticide analyst before they are reported.
- 11.2.1.6 Calculate the sample concentration and on-column concentration of the single component pesticides and surrogates by using the following equations.
- 11.2.1.6.1 Water
- 11.2.1.6.1.1 EQ. 14 Concentration Calculation of Target Compounds in Water Samples

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_o) (V_i)}$$

Where,

$A_x$  = Response (peak area or height) of the compound to be measured.

$\overline{CF}$  = Mean Calibration Factor from the initial calibration (area/ng).

$V_{in}$  = Volume of extract loaded onto GPC column.

$V_{out}$  = Volume of extract collected after GPC cleanup.

$V_t$  = Volume of concentrated extract ( $\mu\text{L}$ ). (If GPC is not performed, then  $V_t = 10,000 \mu\text{L}$ . If GPC is performed, then  $V_t = V_{out}$ .)

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

$GPC = \frac{V_{in}}{V_{out}}$  = Gel Permeation Chromatography factor. (If no GPC is performed,  $GPC = 1.0$ )

$V_o$  = Volume of water extracted (mL). (NOTE: for instrument blanks and sulfur cleanup blanks, assume a 1,000 mL volume).

DF = Dilution Factor. The DF is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed,  $DF = 1.0$ .

The  $\overline{CF}$ s used in Equations 14 - 17 are those from the most recent initial calibration. If the CFs used to determine the linearity of the initial calibration were based on peak area, then the concentration of the analyte in the sample

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must be based on peak area. Similarly, if peak height was used to determine linearity, use peak height to determine the concentration in the sample.

11.2.1.6.1.2 EQ. 15 On-Column Concentration of Water Sample Extract

$$\text{On-Column Concentration (ng/}\mu\text{L)} = \frac{(A_x)}{(\overline{CF}) (V_i)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.6.2 Soil/Sediment

11.2.1.6.2.1 EQ. 16 Concentration of Target Compounds in Soil/Sediment Samples

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_i) (W_s) (D)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_t$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

$W_s$  = Weight of sample extracted (g).

DF = Same as EQ. 14.

D = % dry weight or  $\frac{100 - \% \text{Moisture}}{100}$

GPC = Same as EQ. 14.

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11.2.1.6.2.2 EQ. 17 On-Column Concentration of Soil Sample Extract

$$\text{On-Column Concentration (ng/}\mu\text{L)} = \frac{(A_x)}{(\overline{CF})(V_i)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.7 The lower of the two concentrations calculated for each single component pesticide is reported on Form I. In addition, the concentrations calculated for both the GC columns are reported on Form X, along with a Percent Difference (%Difference) comparing the two concentrations. The Percent Difference is calculated according to Equation 18.

EQ. 18 Percent Difference Between Concentrations on Both GC Columns

$$\%D = \frac{\text{Conc}_H - \text{Conc}_L}{\text{Conc}_L} \times 100$$

Where,

$\text{Conc}_H$  = The higher of the two concentrations for the target compound in question.

$\text{Conc}_L$  = The lower of the two concentrations for the target compound in question.

NOTE: Using this equation will result in Percent Difference values that are always positive.

11.2.1.8 The quantitation of Toxaphene must be accomplished by comparing the heights or the areas of each of the three or four major peaks of in the sample with the CF for the same peaks established during the initial calibration sequence. The concentration of Toxaphene is calculated by using Equations 14 and 16, where  $A_x$  is the area for each of the major peaks. The concentration of each peak is determined and then a mean concentration for the three or four major peaks is determined on each column.

11.2.1.9 The reporting requirement for Toxaphene is similar to that for the single component analytes, except that the lower mean concentration (from three or four peaks) is reported on Form I, and the two mean concentrations reported on Form X. The two mean concentrations are compared by calculating the Percent Difference using Equation 18.

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- 11.1.2.9 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above, and the concentration calculated from the GC/ECD analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form I with one of the laboratory-defined qualifiers ("X", "Y", or "Z"). In this instance, define the qualifier explicitly in the Sample Delivery Group (SDG) Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.
- 11.1.2.10 For GC/MS confirmation of Aroclors, spectra of three characteristic peaks are required for both the sample component and the reference standard.
- 11.1.2.11 The purpose of the GC/MS analysis for the Aroclors is to confirm the presence of chlorinated biphenyls in Aroclors. The GC/MS analytical results for the Aroclors shall not be used for quantitation and the GC/MS results shall not be reported on Form I and Form X. The exception noted in Section 11.1.2.9 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Calculations

11.2.1 Aroclor Concentrations

11.2.1.1 Water

11.2.1.1.1 EQ. 7 Concentration Calculation for Water Samples

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_o) (V_i)}$$

Where,

$A_x$  = Area or height of the peak for the compound to be measured.

$\overline{CF}$  = Mean Calibration Factor from the specific five-point calibration (area/ng).

$V_o$  = Volume of water extracted in mL (Note: for instrument and sulfur blanks assume a volume of 1000 mL).

$V_i$  = Volume of extract injected in  $\mu\text{L}$ . (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column).

$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$ . (If GPC is not performed, then  $V_t = 10000 \mu\text{L}$ . If GPC is performed, then  $V_t = V_{out}$ ).

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DF = Dilution Factor. The DF for analysis of water samples by this method is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

$$\text{GPC} = \frac{V_{\text{in}}}{V_{\text{out}}} = \text{GPC factor. (If no GPC is performed, GPC} = 1.0).$$

$V_{\text{in}}$  = Volume of extract loaded onto GPC column.

$V_{\text{out}}$  = Volume of extract collected after GPC cleanup.

11.2.1.1.2 EQ. 8 On-Column Concentration of Water Sample Extract

$$\text{On-Column Concentration (ng}/\mu\text{L)} = \frac{(A_x)}{(\overline{\text{CF}}) (V_i)}$$

Where,

$A_x$  = Same as EQ. 7.

$\overline{\text{CF}}$  = Same as EQ. 7.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.2 Soil/Sediment

11.2.1.2.1 EQ. 9 Concentration Calculation for Soil Samples

$$\text{Concentration } \mu\text{g}/\text{Kg (Dry weight basis)} = \frac{(A_x) (V_t) (\text{DF}) (\text{GPC})}{(\overline{\text{CF}}) (V_i) (W_s) (D)}$$

Where,

$A_x$ ,  $V_t$ ,  $\overline{\text{CF}}$ , and GPC are as given for water in EQ 7.

$V_i$  = Volume of extract injected in  $\mu\text{L}$ . (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

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$$D = \frac{100 - \% \text{Moisture}}{100}$$

$W_s$  = Weight of sample extracted in g.

DF = Dilution Factor. The DF for analysis of soil/sediment samples by this method is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

11.2.1.2.2 EQ. 10 On-Column Concentration of Soil Sample Extract

$$\text{On-Column Concentration (ng/\mu L)} = \frac{(A_x)}{(\overline{CF})(V_1)}$$

Where,

$A_x$  = Same as EQ. 7.

$\overline{CF}$  = Same as EQ. 7.

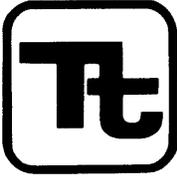
$V_1$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.2 Target Compounds

The quantitation of Aroclors must be accomplished by comparing the heights or the areas of each of a minimum of 3 major peaks of the Aroclor in the sample with the  $\overline{CF}$  for the same peaks established during the specific five-point calibration. The concentration of multi-component analytes is calculated by using Equations 7 and 9, where  $A_x$  is the area for each of the major peaks of the Aroclor. The concentration of each peak is determined and then a mean concentration for a minimum of 3 major peaks is determined on each column.

11.2.2.1 Note that the  $\overline{CF}$ s used for the quantitation of Aroclors are the  $\overline{CF}$ s from the concentration of the specific five-point calibration.

11.2.2.2 The lower mean concentration (from a minimum of 3 peaks) is reported on Form I, and the two mean concentrations reported on Form X. The two mean concentrations are compared by calculating the Percent Difference (%Difference) using Equation 11.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability Tetra Tech NUS, Inc.	
Prepared Chemistry and Toxicology Department	

Subject  
DATA VALIDATION – CLP INORGANICS FOR SOLID AND AQUEOUS MATRICES

Approved  
Tom Johnston *T.E. Johnston*

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## 1.0 PURPOSE

This SOP governs the validation of data generated by inorganics CLP STATEMENT OF WORK (SOW) ILM05.3. As additional inorganic quantification methods are developed, the corresponding validation protocols may be added to this SOP.

## 2.0 APPLICABILITY

The applicability of these validation criteria is described in the appropriate sections below.

## 3.0 PERSONNEL QUALIFICATIONS

The minimum qualifications of persons implementing this SOP are as follow:

- Education – Minimum of a bachelor’s degree in chemistry or related physical/life science.
- Experience requirements include either operational experience with the analytical method or method data review training conducted under the direction of an experienced reviewer and performed on the subject matter data package. A record of the training will not be documented and kept on file but the data validation report produced under training will serve as the record.

## 4.0 INORGANICS (CLP STATEMENT OF WORK (SOW) ILM05.3)

### 4.1 Applicability

This method is applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

The following analytes are commonly determined by this method:

#### **Inductively Coupled Plasma Emission Spectroscopy (ICP)**

Aluminum	Cobalt	Potassium
Barium	Copper	Silver
Beryllium	Iron	Sodium
Cadmium	Magnesium	Vanadium
Calcium	Manganese	Zinc
Chromium	Nickel	

#### **Graphite Furnace Atomic Absorption Spectroscopy (GFAA)**

Antimony	Selenium
Arsenic	Thallium

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### **Cold Vapor Methodology**

Mercury

### **Automated Colorimetric Technique**

Cyanide

#### **4.2 Interferences**

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

#### **4.3 General Laboratory Practices**

The data reviewer must initially verify that a method blank consisting of deionized water was analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

#### **4.4 Sample Preparation**

The data reviewer must initially verify that samples being prepared for ICP, GFAA, and Cold Vapor methodologies are prepared using acid extraction. Samples being prepared for automatic colorimetric technique for Cyanide analysis are prepared using distillation. Additionally the data reviewer must verify that prior to analysis, MS and LCS aqueous and soil samples are spiked with internal standard. The samples are filtered and the extract is ready for CLP analysis.

#### **4.5 Data Overview Prior to Validation**

The data reviewer must initially verify that all CLP Forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required CLP Forms will include:

- a. Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Form 2A.
- b. When reviewing Form 2B, verify that all atomic absorption (GFAA) analytes are present in the CRDL standard at concentrations at the CRDL. Verify that all ICP analytes (with the exceptions of Al, Ba, Ca, Fe, Mg, Na and K) are present in the CRDL standard at concentrations of 2X CRDL.
- c. Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the Form 3 (note that filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).
- d. Verify that all ICP analytes are present in both ICSA and ICSAB solutions. (Note that SOW 3/90 ILM03.0 does not require that antimony, sodium, and potassium be present in these solutions).

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Also verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns. Furthermore, %Rs for solution AB are to be reported to one decimal place on the Form 4.

- e. Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels as noted on pg. E-20 of ILM05.3 Inorganic SOW.
- f. Verify that laboratory duplicate analyses were performed for each matrix. **NOTE:** Field quality control blanks are never to be designated for quality control analyses.
- g. Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis.
- h. The Method of Standard Additions (MSA) Form 8 may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 3.1.3.11 for further details.
- i. Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE:** Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.
- j. Verify that the Form 11 ICP Interelement Correction Factors (Annually) is present.
- k. Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12. Verify that no GFAA analytical results exceed the highest standard used in the associated GFAA calibration.
- l. Verify that the Form 13 Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillations as applicable.
- m. Examine the Form 14s to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. An incorrectly entered "X" flag can lead to reporting errors for the sample and its associated QC. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

#### **4.6 Technical Evaluation Summary**

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (SOP) is intended as proprietary in-house guidance for general inorganic validation practices only.

Evaluate general parameters such as Data Completeness, Overall System Performance, and Detection Limits concurrently with the parameters discussed in the following subsections.

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#### 4.6.1 Holding Times and Sample Preservation Criteria

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

- a. Metals - 6 months; pH <2
- b. Mercury - 28 days; pH <2
- c. Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only; solid samples do not receive preservative, but require maintenance at 4°C.(2°C) during shipment and storage.

#### 4.6.2 Holding Time and Sample Preservation Action

- a. If holding times are exceeded, qualify positive results in affected samples as estimated (J); nondetects (UJ). These results are biased low.
- b. If holding times are exceeded by a factor of more than two times the required time, qualify positive results as estimated (J); qualify nondetects as nondetected rejected (UR). These exceedances are considered to be gross holding time exceedances.
- c. If EPA Regional requirements apply, as in EPA Region III, apply the appropriate bias qualifiers as required; for example, positive results and nondetects as biased low (L) or (UL), respectively.
- d. If samples are received above the required temperature, use professional judgment to qualify the results. Consider the length of time outside the prescribed storage temperature range and other relevant factors.

#### 4.6.3 Initial and Continuing Calibration Requirements Criteria

Verify the following:

- a. **ICP analyses** - must employ a blank and at least one standard. Review initial and continuing calibration Form 2As and associated new data. The initial and continuing calibration %R quality control limits are 90-110%.
- b. **GFAA analyses** - must employ a blank and at least three standards. One of the standards must be at the CRDL. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e., r = 0.995 or greater). The initial and continuing calibration %R quality control limits are 90-110%.
- c. **Mercury analyses** - must employ a blank and at least four standards (r = 0.995 or greater). The initial and continuing calibration %R quality control limits are 80-120%.

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- d. **Cyanide analyses** - must employ a blank and at least three standards ( $r = 0.995$  or greater). **NOTE:** The midpoint standard for cyanide analyses must be distilled; verify this via distillation logs. The initial and continuing calibration %R quality control limits are 85-115%.

#### 4.6.4 Calibration Action

- a. If ICV/CCV %Rs are low, qualify all affected positive results as estimated (J); qualify nondetects as estimated (UJ). In accordance with some USEPA Regional protocol, the (L) and (UL) qualifiers may be used when qualifying results. Bias for these results is low.
- b. If ICV/CCV %Rs are high, qualify all affected positive results as estimated (J); nondetects are not affected. In accordance with some USEPA Regional protocol, the (K) qualifier may be used when qualifying results. Bias for these results is high.
- c. Gross exceedance, as defined by applicable data validation protocol, may require rejection (R) of results.

NOTE: Qualify results of only those samples associated with the noncompliant ICB or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

#### 4.6.5 CRDL Standard Analysis Criteria

Review CRDL Standard Form 2Bs and associated new data. The CRDL Standard analysis %R quality control limits are generally 80-120% for all metals.

#### 4.6.6 CRDL Standard Analysis Action

- a. Generally there is no qualification of data for CRDL %Rs. A comment is noted in the validation letter.
- b. In accordance with some EPA Regional protocol, if CRDL %Rs are high, positive results  $< 2X$  CRDL (Region III) or  $< 3X$  CRDL (Region I) are qualified as biased high (K) or (J), respectively. Note that when using EPA Region I validation guidelines, nondetects will receive qualification based upon high CRDL Standard analysis recovery.
- c. In accordance with some EPA Regional protocol, if CRDL %Rs are low, positive results  $< 2X$  CRDL (Region III) or  $< 3X$  CRDL (Region I) are qualified as biased low (L) or (J), respectively. Nondetected results are qualified as biased low (UL) or (UJ), respectively.

NOTE: The data reviewer need not specify affected samples; common practice is to apply data qualifications "across-the-board" based upon LOE time constraints.

#### 4.6.7 Blank Contamination Criteria

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours whichever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum

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contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all affected samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Professional judgment must be employed when discerning the validity of a concentration present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant review of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

#### 4.6.8 Blank Contamination Action

- a. Qualify as nondetected (U) any positive result within the action level. In accordance with some USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results.
- b. In accordance with some USEPA Regional protocol results are qualified based on negative blank results. Region III requires if any negative blank concentrations are > CRDL then all samples < 5X CRDL are qualified as biased low (L) and nondetects are qualified (UL). Region I requires if any negative blank results are > 2X IDL the nondetected results are qualified as estimated (UJ) and positive results < CRDL are qualified (J).

#### 4.6.9 ICP Interference Check Criteria

Sample Form 4 and associated raw data. Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed 50% (Region III; order of magnitude) of those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\text{Estimated elemental intf.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}] [\text{Conc. in Sample}]}{\text{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

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#### 4.6.10 ICP Interference Check Action

- a. For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. In accordance with some EPA Regional protocol, if ICS %Rs are low, positive results are qualified as biased low (L) and nondetects (UL).
- b. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. In accordance with some EPA Regional protocol, if ICS %Rs are high, positive results are qualified as biased high (K).

**NOTE:** Affected samples include all samples analyzed between the initial and final solutions (or within the eight hour working shift, whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

- c. For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible.
- d. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample.

**(NOTE:** Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

#### 4.6.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Review Spike Sample Recovery Form 5A and associated raw data. Verify that at least one matrix spike was performed for each matrix for a given set of samples within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Refer to ILM03.0, 3/90 Inorganic SOW, Table 3, "SPIKING LEVELS FOR SPIKING SAMPLE ANALYSIS," page 20, Section E, for proper analyte spiking concentrations and requirements. Any deviations from the SOW shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) / Matrix Spike Duplicate (MSD) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant; no actions shall be taken.

#### 4.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. For MS / MSD %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. In accordance with some EPA Regional protocol, if MS/MSD %Rs are low, positive results are qualified as biased low (L).
- b. For MS / MSD %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. In accordance with some EPA Regional protocol, if MS/MSD %Rs are low, positive results are qualified as biased low (L) and nondetects as (UL).
- c. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted. In accordance

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with some EPA Regional protocol, if MS/MSD %Rs are high, positive results are qualified as biased high (K).

#### 4.6.13 Laboratory Duplicate Precision Criteria

Review Laboratory Control Sample Form 6 and associated raw data. Verify that one duplicate sample analysis was performed for each group of samples of a similar matrix within an SDG.

Control criteria used to evaluate aqueous laboratory duplicates are as follows:

- a. A control limit of 20% for relative percent difference when sample and duplicate results are >5X CRDL.
- b. A control limit of 1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL.

Similarly, the following control criteria are generally used to evaluate solid laboratory duplicates:

- a. Control limit of 35% for the relative percent difference when sample and duplicate results are >5X CRDL.
- b. A control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL.

**NOTE:** The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the laboratory duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

#### 4.6.14 Laboratory Duplicate Precision Action

For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples.

**NOTE:** Laboratory duplicate data qualifications shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

#### 4.6.15 Laboratory Control Sample (LCS) Criteria

Review Laboratory Control Sample Form 7 and associated raw data. Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis, and silver and antimony are not subject to quality control criteria. Verify that all solid "found values" fall within the EPA established control limits for soils.

#### 4.6.16 Laboratory Control Sample (LCS) Action

- a. Aqueous
  1. In instances where aqueous LCS %R <80%, qualify positive results as estimated (J) and nondetects as (UJ). In accordance with some EPA Regional protocol, if LCS %Rs are low, positive results are qualified as biased low (L) and nondetects (UL).

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2. If aqueous LCS %R >120, qualify as estimated (J) positive results; nondetects are not compromised by high LCS recovery; thus, no actions are warranted. In accordance with some EPA Regional protocol, if LCS %Rs are high, positive results are qualified as biased high (K).

b. Solids

1. In instances where solid LCS %R found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. In accordance with some EPA Regional protocol, if LCS %Rs are low, positive results are qualified as biased low (L) and nondetects (UL).
2. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results; nondetects are not compromised by high LCS recovery; thus, no actions are warranted. In accordance with some EPA Regional protocol, if LCS %Rs are high, positive results are qualified as biased high (K).

**4.6.17 Method of Standard Additions (MSA) Criteria**

Review MSA Form 8 and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed.

**4.6.18 Method of Standard Additions (MSA) Action**

If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.

NOTE: The "Q" column on the Form 1 of the affected sample should contain an "S" flag for that particular analyte to indicate that the result was obtained using MSA. A "+" flag should also be recorded when the MSA correlation coefficient (r) <0.995. Review the appropriate Form I and amend if necessary.

**4.6.19 ICP Serial Dilution Analysis Criteria**

Review ICP Serial Dilutions Form 9 and associated raw data. Verify that a serial dilution was performed for each matrix and that all ICP analytes are included on the Form 9 with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

**4.6.20 ICP Serial Dilution Actions**

If %D >10% for an analyte, and the corresponding sample concentration is >50x IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of negative interference exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

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NOTE: In accordance with some EPA Regional protocol, the %D should be < 15%.

#### 4.6.21 PA Analysis Run Logs Form 14s Criteria

The Form 14 serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify that GFAA PDS percent recoveries are within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported field sample result or quality control sample result; this can be an area of frequent laboratory error.

#### 4.6.22 PA Analysis Run Logs Form 14s Action

- a. If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample.
- b. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS %R.

#### 4.6.23 Field Duplicate Precision Criteria

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates.

In instances where field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

- a. A control limit of 30% for relative percent difference when sample and duplicate results are >5X CRDL.
- b. A control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL.

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a. A control limit of 50% for the relative percent difference when sample and duplicate results are >5X CRDL.
- b. A control limit of 4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL.

**NOTE:** The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

#### 4.6.24 Field Duplicate Precision Action

For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples.

**NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

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Furthermore, field duplicate data qualifications shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

#### **4.6.25 Further GFAA Evaluations**

It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation or Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

- a. If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

#### **4.7 Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

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## 5.0 REFERENCES

Department of Defense (DoD) Environmental Data Quality Workgroup, 2006. Quality Systems Manual (QSM) for Environmental Laboratories, Final Version 3, January.

EPA540/R-04-004, U.S. EPA, 2004. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October.

US EPA (U.S. Environmental Protection Agency), 2005. Test Methods for Evaluating Solid Waste (SW-846, Third Edition), Physical/Chemical Methods, as amended by Updates I, II, IIA, IIB, III, IIIA, and IIIB, June.

U.S. EPA, 2004. USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration, ILM05.3, March.

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**APPENDIX A  
SAMPLE CALCUATIONS**

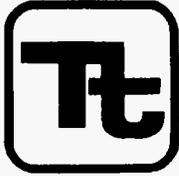
Aqueous Samples:

Verify that the Form I matches the instrument printout.

Soil Samples:

$$\text{Concentration (mg/Kg)} = \frac{A \times D \times E}{B \times C} \times 1\text{L}/1000\text{ml} \times 1000\text{g}/1\text{Kg}$$

- A = Concentration from instrument printout (ug/L)
- B = Initial sample weight (g)
- C = % solids/100
- D = Dilution factor
- E = Final digestion volume (ml)



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>[Signature]</i>	

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT

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## 1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

## 2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

## 3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

## 4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

## 5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

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## 5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

## 5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

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previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

### **5.3 Water Level (Hydraulic Head) Measurements**

#### **5.3.1 General**

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

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Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

### 5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

### 5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.
2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):
  - Well number.
  - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
  - Time and day of the measurement.
  - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

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### 5.3.4 Water Level Measuring Devices

#### Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

#### Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "popping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

#### Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

#### Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

### 5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

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### 5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

### 5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

### 5.5 Health and Safety Considerations

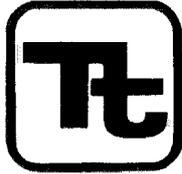
Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

### 6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.







TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Effective Date 06/99	Revision 1
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject  
BOREHOLE AND SAMPLE LOGGING

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## 1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

## 2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

## 5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

### 5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

### 5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.



FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)		FINE-GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size		COARSE-GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	
GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION
OH	Highly organic silty clay with sand and siltstone fragments (see OH).	OH	Highly organic silty clay with sand and siltstone fragments (see OH).	OH	Highly organic silty clay with sand and siltstone fragments (see OH).
CI	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	CI	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	CI	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.
OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.
MI	Inorganic silt of low to medium liquidity, generally silt, silty silt, sandy silt, lean silt.	MI	Inorganic silt of low to medium liquidity, generally silt, silty silt, sandy silt, lean silt.	MI	Inorganic silt of low to medium liquidity, generally silt, silty silt, sandy silt, lean silt.
ML	Inorganic silty clay of low to medium liquidity, generally silty clay, sandy silty clay, silty clay with sand, lean silty clay.	ML	Inorganic silty clay of low to medium liquidity, generally silty clay, sandy silty clay, silty clay with sand, lean silty clay.	ML	Inorganic silty clay of low to medium liquidity, generally silty clay, sandy silty clay, silty clay with sand, lean silty clay.
CL	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	CL	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	CL	Inorganic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.
OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.	OL	Organic clay of low to medium liquidity, generally clay, sandy clay, silty clay, lean clay.
SI	Inorganic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.	SI	Inorganic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.	SI	Inorganic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.
SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.
OL	Organic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.	OL	Organic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.	OL	Organic sand of low to medium liquidity, generally sand, silty sand, sandy sand, lean sand.
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SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	SM	Inorganic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.
OL	Organic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	OL	Organic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.	OL	Organic silty sand of low to medium liquidity, generally silty sand, sandy silty sand, silty sand with sand, lean silty sand.

DENSITY OF GRANULAR SOILS		CONSISTENCY OF COHESIVE SOILS	
TERMINOLOGY	SYMBOL	TERMINOLOGY	SYMBOL
Very Loose	1-2	Very Soft	1-2
Loose	3-4	Soft	3-4
Medium Loose	5-6	Medium Soft	5-6
Loose	7-8	Stiff	7-8
Very Loose	9-10	Very Stiff	9-10
Loose	11-12	Hard	11-12
Very Loose	13-14	Very Hard	13-14
Loose	15-16	Extremely Hard	15-16

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)		ROCK BROKENNESS	
TERMINOLOGY	SYMBOL	TERMINOLOGY	SYMBOL
Very Soft	1-2	Very Soft	1-2
Soft	3-4	Soft	3-4
Medium Soft	5-6	Medium Soft	5-6
Stiff	7-8	Stiff	7-8
Very Stiff	9-10	Very Stiff	9-10
Hard	11-12	Hard	11-12
Very Hard	13-14	Very Hard	13-14
Extremely Hard	15-16	Extremely Hard	15-16

Legend:

- 1" - 3" Standard Sample
- 3" - 6" Unconfined Sample
- 6" - 12" Other Sample, Specific Instructions

Rock Hardness: 1-2, 3-4, 5-6, 7-8, 9-10, 11-12, 13-14, 15-16

Rock Brokenness: 1-2, 3-4, 5-6, 7-8, 9-10, 11-12, 13-14, 15-16

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### 5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inch $\Phi$ -1/2 inch $\Phi$ )" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

### 5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

### 5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

#### 5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

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**FIGURE 2**

**CONSISTENCY FOR COHESIVE SOILS**

<b>Consistency</b>	<b>Standard Penetration Resistance (Blows per Foot)</b>	<b>Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)</b>	<b>Field Identification</b>
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

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Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

#### **5.2.5 Moisture**

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

#### **5.2.6 Stratification**

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

#### **5.2.7 Texture/Fabric/Bedding**

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

#### **5.2.8 Summary of Soil Classification**

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

## FIGURE 3

## BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

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### 5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ( $\text{CaCO}_3$ ). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

#### 5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

**FIGURE 4****GRAIN SIZE CLASSIFICATION FOR ROCKS**

<b>Particle Name</b>	<b>Grain Size Diameter</b>
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

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### 5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

### 5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

### 5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the words "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

### 5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

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The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD  
(After Deere, 1964)

$$RQD \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

### 5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

### 5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

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### 5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

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#### 5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C - Coarse	Lt - Light	Yl - Yellow
Med - Medium	BR - Broken	Or - Orange
F - Fine	BL - Blocky	SS - Sandstone
V - Very	M - Massive	Sh - Shale
Sl - Slight	Br - Brown	LS - Limestone
Occ - Occasional	Bl - Black	Fgr - Fine-grained
Tr - Trace		

#### 5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

##### 5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.
- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.
- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.
- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.
- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5  
COMPLETED BORING LOG (EXAMPLE)



BORING LOG

PROJECT NAME: NSB - SITE BORING NUMBER: SB/MW1  
 PROJECT NUMBER: 9594 DATE: 3/8/96  
 DRILLING COMPANY: SOILTEST CO. GEOLOGIST: SJ CONTI  
 DRILLING RIG: CME-55 DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			U S C S *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 e 0800	0.0 2.0	7 6 10	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME ROCK FR. - TR BRICKS (FILL)	SM	MOIST SL. ORG. ODOR FILL TO 4'±	5	0	0	0
S-2 e 0810	4.0 6.0	5 7 8	2.9/2.0	4.0	M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
S-3 e 0820	8.0 10.0	6 8 17 16	1.9/2.0	7.0 8.0	DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER: 7'±	0	0	0	0
S-4 e 0830	12.0 14.0	7 6 8	1.6/2.0	12.0	STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	5	-	-
	15.0			15.0					AUGER REF @ 15'				
	16.0			16.0	M HARD	BRN	SILTSTONE	VER	WEATHERED				
	17.0			17.0					LO *JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
	18.0			18.0					LOSING SOME				
	19.0			19.0	HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H <sub>2</sub> O @ 17'± SET TEMP 6" CAS TO 15.5				
	20.0			20.0									
	21.0			21.0					SET 2"Ø PVC SCREEN 16-25	0	0	0	0
	22.0			22.0					SAND 14-25				
	23.0			23.0					PELLETS 12-14				

\* When rock coring, enter rock brokenness.  
 \*\* Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.  
 Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ± • 1-20Z  
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z  
NIX CORE IN BEDROCK RUN (1) = 25 min, RUN (2) = 15 min Drilling Area Background (ppm):   
 Converted to Well: Yes  No  Well I.D. #: MW-1

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- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
  - Particle shape - flat, elongated, or flat and elongated.
  - Maximum particle size or dimension.
  - Water level observations.
  - Reaction with HCl - none, weak, or strong.
- Additional comments:
  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

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- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.
- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

### 5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.
- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.
- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.
- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.
- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.
- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.
- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:
  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.
- Remarks at the bottom of Boring Log shall include:
  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.
- As a final check the boring log shall include the following:
  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.
  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

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### 5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

### 5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

### 6.0 REFERENCES

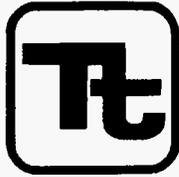
Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

### 7.0 RECORDS

Originals of the boring logs shall be retained in the project files.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Effective Date 09/03	Revision 3
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject  
GROUNDWATER MONITORING WELL INSTALLATION

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## 1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

## 2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

## 3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

## 4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

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## 5.0 PROCEDURES

### 5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

### 5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

#### 5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

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The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

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Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

### 5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

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### **5.2.3 Annular Materials**

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium-to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

### **5.2.4 Protective Casing**

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

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A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized lid is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

### 5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

#### 5.3.1 **Monitoring Wells in Unconsolidated Sediments**

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

#### 5.3.2 **Confining Layer Monitoring Wells**

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

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installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

**5.3.3 Bedrock Monitoring Wells**

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

**5.3.4 Drive Points**

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

**5.3.5 Innovative Monitoring Well Installation Techniques**

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

**5.4 Well Development Methods**

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

**5.4.1 Overpumping and Backwashing**

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

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remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

#### **5.4.2 Surging with a Surge Plunger**

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

#### **5.4.3 Compressed Air**

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

#### **5.4.4 High Velocity Jetting**

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

### **6.0 RECORDS**

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

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space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

## 7.0 REFERENCES

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Cosby, and J. Fryberger, 1981. Manual of Groundwater Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

**ATTACHMENT A**

**RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)**

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

- |    |                     |   |                  |
|----|---------------------|---|------------------|
| 1  | Teflon <sup>®</sup> | 5 | Lo-Carbon Steel  |
| 2  | Stainless Steel 316 | 6 | Galvanized Steel |
| 3. | Stainless Steel 304 | 7 | Carbon Steel     |
| 4  | PVC 1               |   |                  |

\* Trademark of DuPont

**RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)**

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton <sup>®*</sup>	Silicone	Neoprene	Teflon <sup>®*</sup>
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

- |    |                        |   |                        |
|----|------------------------|---|------------------------|
| 1  | Teflon <sup>®</sup>    | 5 | PE Conventional        |
| 2  | Polypropylene (PP)     | 6 | Plexiglas/Lucite (PMM) |
| 3. | PVC Flexible/PE Linear | 7 | Silicone/Neoprene      |
| 4  | Viton <sup>®</sup>     |   |                        |

\* Trademark of DuPont

Source: Barcelona et al., 1983

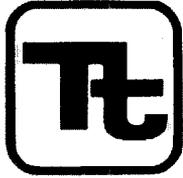
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**ATTACHMENT B**

**COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION**

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

\* See also Attachment A.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
RESISTIVITY AND ELECTROMAGNETIC INDUCTION

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## 1.0 PURPOSE

The purpose of this guideline is to provide a general description of, and technical management guidance on, the use of Resistivity and Electromagnetic Induction (Ground Conductivity) surveys during hazardous waste site investigations.

## 2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability, and implementability of geophysical methods used during hazardous waste site investigations to determine subsurface resistivity or conductivity. Measurements of subsurface conductivity or resistivity can be used to determine the presence and approximate extent of subsurface contaminants, buried drums, and metal containers. In addition, the depth to the water table, and structural characteristics of the subsurface environment can be determined.

This document is intended to help develop a sufficient understanding of each method to assist in proper work plan development and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation. The highly specialized nature of these methods requires inclusion of project-specific, site-specific, and subcontractor-specific information prior to development of detailed operating procedures. Specialized expertise during both planning and execution of these geophysical methods is also required.

The description focuses on methods and equipment that are readily available and typically applied; it is not intended to provide a complete discussion of the state of the art.

## 3.0 GLOSSARY

Apparent Conductivity - The quantity measured during an electromagnetic induction survey; proportional to the actual conductivities of subsurface materials.

Apparent Resistivity - The quantity actually deduced during a resistivity survey; proportional to the actual resistivities of subsurface materials.

Conductivity - Intrinsic property of a substance, equal to the reciprocal of resistivity.

Current - The quantity of charge transmitted per unit time.

Electromagnetic (EM) Induction Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Potential - Intrinsic property of electrical fields, equating to the ability to do work. A potential field can induce a potential difference (voltage) between two electrodes.

Resistivity - Intrinsic property of a substance, equal to the resistance of a body multiplied by its cross-sectional area and divided by its length.

Resistivity Survey - A geophysical exploration method whereby an electrical current is transmitted into the ground and the resultant potential field is measured to deduce the apparent subsurface resistivity.

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#### 4.0 RESPONSIBILITIES

Project Manager - responsible for the scoping of geophysical surveys during development of the Work Plan, with the help of the Field Operations Leader, site geologist, and site geophysicist.

Field Operations Leader (FOL) – Responsible for overall management and coordination of the field work.

Site Geophysicist - as a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of these techniques for providing necessary data. Field work for these surveys is supervised by the site geophysicist.

#### 5.0 PROCEDURES

##### 5.1 Electromagnetics

The electromagnetic induction (EM) method provides a means of measuring the electrical conductivity of subsurface soil, rock, and groundwater. Electrical conductivity is a function of the type of soil and rock, its porosity, its permeability, and the fluid composition and saturation. In most cases the conductivity of the pore fluids will be responsible for the measurement. Accordingly, the EM method applies both to assessment of natural geohydrologic conditions and to mapping of many types of contaminant plumes. In addition, trench boundaries, buried wastes, drums, and utility lines can be located with EM techniques.

##### 5.1.1 Applicability

Although EM is not a definitive technique, it is useful for several reasons. First, an EM survey can be conducted over an entire site very quickly. In addition, EM methods are generally inexpensive, even for coverage of large areas. Often, 100 acres or more may be surveyed in just a few days time (depending on desired detail). More importantly, EM data can be used to focus the more expensive phases of an investigative project, potentially resulting in a large cost savings. For example, rather than drilling several dozen monitoring wells while searching for groundwater contamination, an EM conductivity unit may be used to survey for a conductive (or resistive) plume. Several EM survey lines may be run to provide definition of the plume and an indication of its source area, reducing the number of exploratory wells required and potentially resulting in better well placement providing a potentially significant cost savings. Another reason why EM should be considered is to fill in data gaps and to reduce the risk of missing a facet of the investigation, such as the presence of undetected refuse trenches, buried drums, or changing hydrologic conditions.

Electromagnetic methods may be used in many situations for a variety of purposes. The following list includes major uses related to investigations of hazardous waste sites:

- Defining the location of a contaminant plume [This could lead to the identification of downgradient receptors, source areas, and flow directions if the conductivity of the plume (target) is distinct in comparison to the host (background), hydrogeologic setting.]
- Locating buried metal objects (e.g., drums, tanks, pipelines, cables, monitoring wells).
- Addressing the presence or location of bedrock fault/fracture systems (This is important for identification of groundwater preferential pathways in bedrock.)
- Mapping grain size distributions in unconsolidated sediments.

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- Mapping buried trenches and contaminated fill materials.
- Mapping saltwater intrusion.
- Defining lithological (unit) boundaries.
- Determining the rate of plume movements by conducting multiple surveys over time.

The above list is only partial; in fact, EM methods may be used wherever a significant change in conductance can be measured. EM should be considered for use when a suspected target is anticipated to have a conductivity significantly different from background values. Factors such as cost, site-specific conditions, and equipment availability should also be evaluated before deciding to proceed with an EM survey.

### 5.1.2 General

#### 5.1.2.1 Objectives

The site geophysicist should evaluate the objectives of the site investigation in light of EM capabilities. If the purpose of the site investigation is to confirm the presence of contaminants with minimal effort, EM methods may provide too much detail and no direct evidence. Direct methods, on the other hand, such as installing monitoring wells with limited sampling, may be more suitable. If a site is to be characterized in detail and if assessment of hydrogeologic conditions and identification of all source areas, plumes, and receptors are a priority, then EM (and other geophysical methods) may be a more cost-effective way of selecting strategic locations for monitoring wells, directing test pit operations, efficiently selecting sampling points, and providing information between site sampling points.

#### 5.1.2.2 Existing Data

If EM equipment is identified as a viable alternative for providing the type of information desired, the user should further evaluate the equipment to determine whether it is appropriate for use under the conditions found at a particular site. Evaluation of existing data can identify problems that may be encountered in the field:

- Variations in hydrogeologic conditions (e.g., varied water table conditions or changes in rock or sediment) can result in a conductivity range that envelopes the response of the target (e.g., plume) and effectively masks or blocks out any signals.
- Scattered, near-surface metal may mask buried targets such as drums or trenches.
- Anthropogenic features such as overhead powerlines, buried metallic pipes, etc., may decrease the signal to noise ratio making the technique ineffective.

An analysis of the site history might more closely define a survey area, thereby cutting survey costs by reducing the size of the survey. Deep targets may be out of the penetration range for many EM units, and specialized equipment may be required. It is difficult for EM systems to detect a groundwater contaminant plume through 100 feet of unsaturated overburden. A site reconnaissance should be conducted to identify other site conditions that may affect the data. Drastic topography changes can affect the quality of EM data obtained with some systems, and this possibility should be considered at each site.

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### 5.1.3 Survey Design

Once the EM survey objectives have been defined, existing information has been reviewed, and reconnaissance of the site has been conducted, attention should be given to the design of the geophysical survey. The detail required of an EM survey is a primary factor in designing and planning fieldwork. If the purpose of performing EM work on site is to define a large geologic feature, then a grid using a wide (100- to 1,000-foot) line spacing may be needed. Some instruments are capable of providing a continuous data profile, which makes it less likely to miss small conductors than the typical discrete measurement EM instruments. The importance of designing and implementing a grid system tied into existing "permanent" features (such as roads and buildings) cannot be overstated. This permanent feature will allow the grid to be re-established in the field at a later time in order to place drill holes and monitoring wells. Furthermore, additional surveys may be conducted on the site over time using other geophysical techniques or the same technique to provide an indication of plume movement. These surveys will help in orienting maps and diagrams that are produced later and in defining targets.

#### 5.1.3.1 Background Noise

Background noise can be a significant factor in the success of an EM survey. Evaluation of existing data and a site reconnaissance will help to identify the probable background noise level. A high noise level can make interpretation difficult and may actually cause an anomaly to be overlooked. It is difficult to delineate a conductive contaminant plume contained in overburden that has a wide natural variation in conductivity.

Noise can be divided into two groups: (1) natural, such as changing grain size distributions, steeply dipping strata, undetected mafic dikes, karstic topography, unexpected fault zones; and (2) cultural, such as power lines, houses, railroads, surface metal debris, cars, radio transmission towers, and other metal objects which are not intended to be located by the survey. Some instruments are more sensitive to certain types of noise sources than others. Because there is little published information on this subject, appropriate experience is essential.

#### 5.1.3.2 Limitations

All EM instruments have varying limitations with regard to sensitivity and penetration. Published references, operator's manuals, and field experience should be used to evaluate instrumentation versus capability. Table 1 lists several commercially available instruments along with operator requirements and productivity estimates.

#### 5.1.3.3 Instrumentation

Table 2 provides guidance for EM equipment selection. These instruments may not be suitable to specific site conditions and investigation objectives. The decision to use a specific instrument is dependent upon site factors.

Electromagnetic techniques have also been adapted for downhole applications. These techniques can be useful in defining the vertical extent of a contaminant zone. Some systems work inside polyvinyl chloride (PVC) or Teflon monitoring well casings (see SOP GH-3.5, Borehole Geophysical Surveys).

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**TABLE 1**

**COMMON EM AND RESISTIVITY EQUIPMENT**

Instrument	Manufacturer	Minimum No. of Operators	Typical Daily Line Miles (50-ft readings)	Notes
EM-16-R	Geonics	2	2	2
EM-38	Geonics	1	3 - 4	2
EM-31-D	Geonics	1	3 - 4	2
EM-34-3	Geonics	2	2	2
PROTEM47P	Geonics	2	0.25	1
EM-61	Geonics	1	3 - 4	3
T-VLF	Iris Inst.	1	3 - 4	2
CEM	Crone	2	2	1
Max Min II	Apex	2	3	1
Syscal Resistivity Meter	Iris Inst.	2	0.5	2

Notes:

1. Primarily useful for geologic features only.
2. Useful for geologic and cultural features.
3. Primarily useful for mapping buried metals.

Designations such as EM-31 or EM-61 are the manufacturer's model numbers and do not imply equipment complexity or capability.

Table 2  
Application Guidelines for Equipment Use

Application	Technique and Instrumentation									
	VLF	VLF Resistivity	Frequency Domain EM	Time Domain Soundings	Time Domain EM Metal Detection	Resistivity Sounding	Resistivity Profiling	Azimuthal Resistivity Surveying		
Archeological Studies			X							
Locate Single Buried Steel Drum			X							
Locate a Cluster of Buried Steel Drums			X							
Delineate a Landfill			X							
Delineate Contaminated Soil and Fill			X				X			
Map Groundwater Contamination		X	X	X		X				
Locate Metallic Pipelines and Utilities	X		X		X					
Delineate USTs			X		X					
Map Stratigraphy		X	X				X			
Map Joints and Faults	X		X					X		

Notes:

	Not applicable
X	Sometimes applicable but other techniques may be more cost effective
X	Often Applicable and cost effective

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## 5.2 Electrical Resistivity

Electrical resistivity surveys provide information about the subsurface distribution of the ground resistivity. The information can be used to infer groundwater quality, lithologic, and geologic information. Both horizontal and vertical changes in ground resistivity can be mapped by resistivity surveys. In practice, resistivity surveys are mostly used to determine the vertical resistivity changes. Lateral resistivity changes are more easily mapped by electromagnetic surveys.

### 5.2.1 **Applicability**

Electrical resistivity (ER) data are subject to interpretation; therefore, ER field results should be checked periodically and confirmed by direct methods, such as sampling or drilling.

Although ER is not a definitive technique, the data are useful for several reasons. Typical productivity with conventional resistivity equipment is several thousand line-feet per day. This high productivity rate allows a large amount of useful data to be collected in a relatively short period of time. For example, rather than drilling several dozen monitoring wells or test borings to develop a complete picture of the site stratigraphy and structure, a few wells can be drilled (for control) and information about the rest of the site can be obtained by using resistivity methods. Method integration such as this can reduce the amount of time and the costs required for a project.

Resistivity methods may be used in a wide array of situations and for a variety of purposes. The following is a partial list of major uses related to investigations of hazardous waste sites:

- Definition of a contaminant plume. (This could lead to the identification of downgradient receptors and source areas.)
- Waste pit delineation.
- Definition of bedrock fault/fracture systems.
- Water table mapping (for contour maps).
- Stratigraphic mapping of soil layers (particularly useful in overburden, discriminating clays from sands and establishing their thicknesses).
- Defining bedrock topography (valleys).

Resistivity methods may be used whenever the feature to be mapped has a contrasting resistivity with the background material.

### 5.2.2 **General**

Electrodes are typically arranged in one of several patterns, called electrode arrays, depending on the desired information. Electrical resistivity techniques can determine the vertical subsurface resistivity distribution beneath a point. In this type of survey, called vertical electrical soundings, the electrode array is expanded systematically and symmetrically about a point. For each set of electrode spacings, apparent resistivity is determined from measurements of potential and input current. The resultant plot of apparent resistivity versus electrode spacing is interpreted to provide the subsurface resistivity with depth distribution at that one particular point. Examples of three common arrays are given in Figure 1. The Wenner and Schlumberger arrays are somewhat more common than the Dipole-Dipole and other arrays.

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These arrays (Wenner, Schlumberger) start with a small electrode spacing that is increased to permit deeper penetration for sounding.

The manner in which the apparent resistivity changes with the electrode separation can be used to determine formation conductivity and layer thickness. To increase accuracy, the user should evaluate the interpretation of resistivity data against the existing subsurface information. With any set of apparent resistivity readings, a number of solutions are possible, so existing data must be used to select the one that fits best. A formation resistivity may be assigned, but without geological control the material is not known. Resistivity electrode arrays can also be used with constant inner-electrode spacing to develop a lateral picture of the site through profiles. Stratigraphic control is even more important when mapping lateral changes with constant electrode spacings, because layer thickness changes alone can cause changes in apparent resistivity. The desired resolution is a major factor in deciding how closely to space measurements for a given survey.

In practical application, a resistivity survey target (such as a plume or clay lens) should have a resistivity contrast (positive or negative) of 20 percent from background. This change in resistivity should be 50 percent or more to provide proper detection and delineation. For example, if a resistivity survey were conducted to delineate a groundwater contaminant plume (in overburden) with a resistivity of 200 ohm-meters, a background, saturated overburden resistivity of over 400 ohm-meters (for a conductive plume) or under 100 ohm-meters (for a resistive plume) would probably allow detection of the plume, providing other factors (such as depth) are not detrimental.

When depth sounding, resolution of individual layers has an accuracy generally around 20 percent; accuracy can be substantially more or less depending on the site conditions and operator expertise. Vertical resistivity sounding is usually less accurate than seismic refraction work, which is often conducted within a 10 percent error tolerance. However, geologic units may be distinguishable (by geophysics) only with the use of resistivity methods at some sites.

### 5.2.3 Survey Design

Data can be collected at randomly located stations or along survey lines. If vertical electrical soundings are performed to obtain resistivity changes with depth, then the soundings are positioned where the information is most useful. If measurements are made to map lateral resistivity changes, then the survey is best performed on a grid or on survey lines. The station spacing will be determined from the target size.

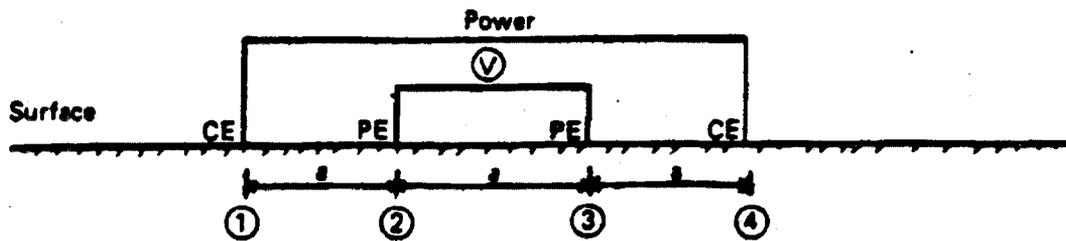
#### 5.2.3.1 Background Noise

Evaluation of existing data and a site reconnaissance will help to identify the possible background noise level. A background high noise level can make interpretation difficult and may mask an anomaly. It would be difficult to delineate a slightly conductive contaminant plume contained in overburden that has wide natural variations in conductivity. Noise can be divided into two groups: natural, such as discontinuous clay layers, undetected mafic dikes, karstic topography, unexpected fault zones, variable water table, and lightning; and cultural, such as power lines, railroad tracks, and radio transmission towers. Since there is little published information on instrument noise sensitivity, appropriate experience is essential.

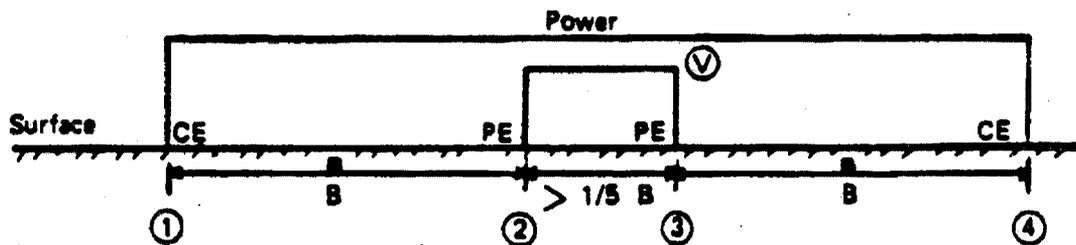
FIGURE 1

## EXAMPLES OF COMMON ER ARRAYS

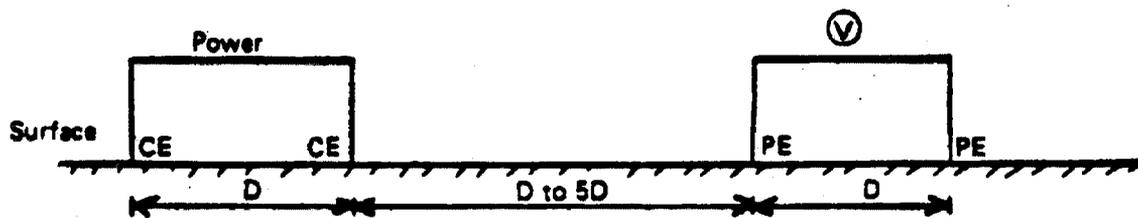
## WENNER ARRAY



## SCHLUMBERGER ARRAY



## DIPOLE-DIPOLE ARRAY



- ① Electrode Number
- PE Potential Electrode
- CE Current Electrode
- Ⓥ Voltmeter

Source: Based in part on W. M. Telford et al., Applied Geophysics, 1976, and R. E. Sheriff, Encyclopedic Dictionary of Exploration Geophysics, 1984.

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#### 5.2.3.2 Depth of Investigation

As a rule of thumb when a lateral resistivity survey is being conducted, the array should be spaced four or five times the distance from the ground surface down to the desired target. For vertical sounding, this suggested spacing should be about ten times the anticipated target depth. These suggestions should be used only as general guidance.

#### 5.2.4 **Miscellaneous Considerations**

##### 5.2.4.1 Instrumentation

For most shallow work at hazardous waste sites, most resistivity systems will suffice. Generally, equipment capability becomes important only when the desired investigative depth exceeds 70 to 100 feet. Larger power sources are needed to provide a measurable electrical potential with a wider electrode spacing. Some newer resistivity units are capable of electronic data storage, and other features. Often, the peripheral capabilities of an ER system may be the deciding factor when purchase is considered.

Borehole resistivity equipment has been used (in fluid filled uncased boreholes) to determine relative formation porosity and other factors. For more information on this equipment, the reader should refer to the borehole geophysics subsection of this compendium.

##### 5.2.4.2 Calibration

ER equipment requires calibration, either in the field or in the laboratory; dated records of this calibration should be kept in the equipment management file and in the appropriate project file. Calibration is used to establish the reliability and accuracy of the equipment; calibration typically includes an internal circuit check or actual field trials (e.g., tests over a known target). Equipment that historically exhibits fluctuations in calibration should not be used. The equipment serial number should be recorded on the calibration records. If the manufacturer recalls equipment, this fact should be explained and documented for instrument maintenance in the proper file. The current source and potentiometer must be calibrated on any type of resistivity equipment. The instrument's current source may be calibrated by placing a reference ammeter in series with the electrode cables. The reading obtained on the reference ammeter is compared with the value read from the instrument's current source ammeter. The current source ammeter is then adjusted accordingly.

The potentiometer is calibrated by either of two methods. The preferred field method, which is similar to the calibration of the current source, is done by comparing the instrument's indicated potential to that potential measured with an independent voltmeter. An alternative means of calibration, which can be performed in the laboratory, involves placing a precision resistor of a known value in series with the current load. A potentiometer is then placed across the resistor. The potential measured should be equal to the product of the known resistance and indicated current.

##### 5.2.4.3 Data Reduction

Direct current (DC) and low frequency alternating current (AC) resistivity meters measure two values:

- (1) the amount of current injected into the subsurface via the current and sink electrodes; and,
- (2) the potential (voltage) between two or more electrodes that are separated a known distance apart (a-spacing).

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The resistance (ohms) can be calculated from these known values using Ohm's law.

Ohm's law states the following:

$$v = ir \text{ or rearranged : } r = v/i$$

where:

v	=	voltage (volts)
i	=	current (amperes)
r	=	resistance (ohms)

Resistivity is a measurement of resistance across a distance and over a cross-sectional area. Resistivity is calculated by the following equation that assumes that the resistor is a rectangular block:

$$R = ra/L$$

where:

R	=	resistivity (commonly expressed in units of ohm-meters or ohm-feet)
r	=	resistance (v/i, in units of ohms)
a	=	cross-sectional area of the resistor
L	=	length of the resistor

When calculating resistivity in the three-dimensional earth, the resistor becomes hemispherical. Ohm's law becomes the following:

$$R = (r * 2\pi d^2)/d \text{ or } R = (r * 2\pi d)$$

where:

r	=	resistance (ohms)
$2\pi d^2$	=	surface area of a hemisphere
d	=	diameter of sphere

The value for the diameter of the sphere is analogous to the distance between the potential electrodes. This distance is referred to as the "a-spacing" when using the Wenner resistivity array and is measured in units of meters or feet.

Apparent resistivity ( $\rho$ ) is determined from a single measurement because the earth is heterogeneous, and horizontal layers of earth act as a circuit with resistors in parallel. True resistivity is found by performing a resistivity sounding. Resistivity soundings are produced by determining the apparent resistivity across successively increasing electrode separation distances. The results of the resistivity soundings are plotted on semi-log paper. Apparent resistivity values are plotted on a linear scale and the potential electrode separation distance is plotted on a log scale for the Wenner array. The resulting curve is then matched to a master set of curves to determine true resistivity. Alternatively, a mathematical model can be used to determine the true resistivity of each layer that is within the depth of investigation of the instrument.

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Most modern resistivity meters contain a data logger that records voltage, amperes, and a-spacing values. These resistivity meters often contain processors that calculate apparent resistivity ( $\rho$ ) and monitor levels of signal noise that are due to anthropogenic features or changes in geology across the measurement area. Apparent resistivity values calculated with these instruments should also be spot checked to ensure the data quality.

Several forward and inverse modeling software packages (e.g., RESIX™) exist that can be used to calculate the true resistivity of the subsurface layers. These computer models require some knowledge of the geology of the site where data collection occurs. Electric log records or estimates of the corresponding geologic layers' electrical resistivity are also required for calibration of the computer model. Typical resistivity values for various types of soil and rock are published in most geophysical textbooks.

## 6.0 REFERENCES

### 6.1 Electromagnetic Induction

#### 6.1.1 Electromagnetic (EM) Theory and Interpretation Textbooks

Grant, F. S., and G. F. West. Interpretation Theory in Applied Geophysics. McGraw-Hill Book Company. 1955.

Griffiths, D. H., and R. F. King. Applied Geophysics for Geologists and Engineers. Pergamon Press. 1981.

McNeill, J. D. "Electrical Conductivity of Soils and Rock." Technical Note No. 5. Mississauga, Canada: Geonics Limited. 1980.

McNeill, J. D. "Electromagnetic Terrain Conductivity Measurement at Low Induction Numbers." Technical Note No. 6. Mississauga, Canada: Geonics Limited. 1980.

McNeill, J. D. "Interpretative Aids for Use with Electromagnetic (Non-Contacting) Ground Resistivity Mapping." Paper presented at European Association of Exploration Geophysicists Annual Meeting. Hamburg, Germany. 1979.

Parasins, D. S. Principles of Applied Geophysics (3rd Edition). Chapman and Hall Publishers. 1979.

Telford, W. M., L. P. Geldard, R. E. Sheriff, and D. A. Keys. Applied Geophysics. Cambridge University Press.

Wait, J. R. "A Note on the Electromagnetic Response of a Stratified Earth." Geophysics, Vol. 21, pp. 382-385.

Wait, J. R. Geo-Electromagnetism. Academic Press. 1982.

Zohdy, Adel A.R. "The Use of Dar Zarrouk Curves in the Interpretation of Vertical Electrical Sounding Data." Bulletin 1313-D, USGS. 1979.

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### 6.1.2 EM General Manuals

Benson, R. C., R. A. Glaccum, and M. F. Noel. "Geophysical Techniques for Sensing Buried Wastes and Waste Migration." U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada. 1983.

Jordan, T. E., and D. Constantini. "The Use of Non-Invasive Electromagnetic (EM) Techniques for Focusing Environmental Investigations." The Professional Geologist. Vol. 32, No. 7, 1995.

Jordan, T. E., et al. "The Use of High Resolution Electromagnetic Methods for Reconnaissance Mapping of Buried Wastes." The Proceedings of the Fifth National Outdoor Action Conference, Surface Geophysical Methods, National Water Well Association, Las Vegas, NV. 1991.

McNeill, J. D. "Electromagnetic Resistivity Mapping of Contaminant Plumes." Presented at the National Conference on Management of Uncontrolled Hazardous Waste Sites--contact HMCRI. Silver Spring, Maryland.

Rudy, R. J., and J. A. Caoile. "Utilization of Shallow Geophysical Sensing at Two Abandoned Municipal/Industrial Waste Landfills on the Missouri River Floodplain." Ground Water Monitoring Review. Fall issue, 1984.

Slaine, D. D., and J. P. Greenhouse. "Case Studies of Geophysical Contaminant Mapping at Several Waste Disposal Sites." Presented at the NWWA Second National Symposium on Aquifer Restoration and Ground Water Monitoring. Columbus, Ohio. 1982.

Steward, M. T. "Evaluation of Electromagnetic Methods for Rapid Mapping of Salt-Water Interfaces in Coastal Aquifers." Groundwater, Vol. 20. September-October 1982.

### 6.1.3 Manufacturers

Aerodat Limited  
3883 Nashua Drive  
Mississauga, Ontario L5V 1R3  
416/671-2446 (airborne EM systems)  
systems)

Phoenix Geophysics Limited  
200 Yorkland Boulevard  
Willowdale, Ontario M2J 1R5  
416/493-6350 (surface EM  
EM

Crone Geophysics Limited  
3607 Wolfedale Road  
Mississauga, Ontario L5C 1V8  
416/270-0096 (surface EM systems)  
systems)

Scintrex  
222 Snidercroft Road  
Concord, Ontario L4K 1B5  
416/669-2280 (surface EM  
EM

Geonics Limited  
1745 Meyerside Drive  
Mississauga, Ontario L5T 1C5  
416/676-9580 (borehole and surface EM systems)

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## 6.2 Electrical Resistivity

### 6.2.1 Electrical Resistivity (ER) Theory and Interpretation Textbooks

Griffith, D. H., and R. F. King. Applied Geophysics for Geologists and Engineers. Pergamon Press. 1981.

Grant, F. S., and F. G. West. Interpretation Theory in Applied Geophysics. McGraw-Hill. 1965.

Telford, W. M., et al. Applied Geophysics. Cambridge University Press. 1976.

### 6.2.2 Journals

Zohdy, A. A. R. "Automatic Interpretation of Schlumberger Sounding Curves Using Modified Dar Zarrovk Functions." U.S. Geological Survey Bulletin, 1313 E., Washington, D.C. 1975.

### 6.2.3 ER General Manuals

Benson, R. D., R. S. Glaccum, and M. R. Noel. Geophysical Techniques for Sensing Buried Wastes and Waste Migration. U.S. Environmental Monitoring Systems Laboratory. Las Vegas, Nevada. 1983.

Costello, R. L. Identification and Description of Geophysical Techniques. Prepared by D'Appolonia Corporation for the U.S. Army Toxic and Hazardous Materials Agency. Aberdeen Proving Ground, Maryland. 1980.

Greenhouse, J. P. Surface Geophysics in Contaminant Hydrogeology. Manual for the Hydrology Field School through the University of Waterloo, Ontario, Canada. 1982.

Peffer, J. R., and P. G. Robelen. Affordable: Overburden Mapping Using New Geophysical Techniques. Pit and Quarry. August 1983.

Technos, Incorporated. Application Guidelines for Selected Contemporary Techniques for Subsurface Investigations. (No publication date given.)

### 6.2.4 ER Case Histories and Examples Journals

Bradbury, K. R., and R. W. Taylor. "Determination of the Hydrologic Properties of Lakebeds Using Offshore Geophysical Surveys." Ground Water, Vol. 22, No. 6. 1984.

Evans, R. B., and G. E. Schweitzer. "Assessing Hazardous Waste Problems." Environmental Science Technology, Vol. 18, No. 11. 1984.

Pennington, D. "Selection of Proper Resistivity Techniques and Equipment for Evaluation of Groundwater Contamination." Presented at the NWWA Conference on Surface and Borehole Geophysical Methods in Groundwater Investigation. Fort Worth, Texas. February 1985.

Ringstad, C. A., and D. C. Bugenig. "Electrical Resistivity Studies to Delimit Zones of Acceptable Ground Water Quality." Ground Water Monitoring Review. Fall 1984.

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Taylor, R. W., and A. H. Fleming. "Characterizing Jointed Systems by Azimuthal Resistivity Surveys." Ground Water. V. 26, No. 4, 1988.

Underwood, J. W., K. J. Laudon, and T. S. Laudon. "Seismic and Resistivity Investigations near Norway, Michigan." Ground Water Monitoring Review. Fall 1984.

### 6.2.5 Manufacturers

ABEM-Atlas Copco  
Distributed by Geotronic Corp.  
10317 McKalla Place  
Austin, Texas 78758

Phoenix Geophysics Limited  
200 Yorkland Boulevard  
Willowdale, Ontario M2J 1R5

Bison Instruments, Inc.  
570-8 West 36th Street  
Minneapolis, Minnesota 55416

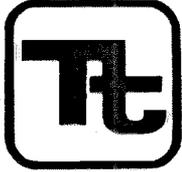
Scintrex Limited  
222 Snidercroft Road  
Concord (Toronto), Ontario L4K 1B5

BRGM-Syscal  
Distributed by EDA Instruments  
5151 Ward Road  
Wheat Ridge, Colorado 80033

### 7.0 RECORDS

The following information will be recorded in the field log book.

- Date
- Equipment operators
- Name and project number of site
- Position and instrument readings
- Position-specific information



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
MAGNETIC AND METAL DETECTION SURVEYS

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## 1.0 PURPOSE

The purpose of this guideline is to provide a general description of, and technical management guidance on, the use of Magnetic and Metal Detection Surveys for site investigations.

## 2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability, and implementability of standard geophysical methods used during site investigations to determine site features related to magnetic anomalies and buried metal. This document is intended to be used by the project manager, field operations leader, or site geologist to develop a sufficient understanding of each method and to assist in proper work plan development and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation. The highly specialized nature of the subject geophysical methods requires inclusion of project-specific, site-specific, and subcontractor-specific information prior to development of detailed operating procedures, during both planning and execution.

The description focuses on methods and equipment that are readily available and typically applied; it is not intended to provide a complete discussion of the state of the art.

## 3.0 GLOSSARY

Magnetic Survey -- A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Magnetic Susceptibility -- Property of a material corresponding to its ability to distort an applied magnetic field.

Magnetometer -- A device used for precise and sensitive measurements of magnetic fields.

Magnetometry -- The science of measuring variations in the earth's magnetic field.

Metal detection -- A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer -- A magnetometer equipped with two sensors that are vertically separated a fixed distance apart. It is best suited to map near surface features and is less susceptible to deep geologic features.

## 4.0 RESPONSIBILITIES

Project Manager -- responsible for scoping the magnetic or metal detection surveys during development of the Work Plan with the help of the site geologist and site geophysicist.

Field Operations Leader (FOL) -- responsible for overall management and coordination of the field effort.

Site Geophysicist -- central role in determining the technique used for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical

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specialists and other personnel as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialists.

## **5.0 PROCEDURES**

### **5.1 Description of Methods**

#### **5.1.1 Theory and Principles of Operation**

##### **5.1.1.1 Magnetometry**

Materials subject to a magnetic field will develop an induced magnetization, proportional to the applied magnetic field and the magnetic susceptibility of the material.

Induced magnetization in an object produces a local magnetic field which either reinforces (positive magnetic susceptibility) or reduces (negative susceptibility) the external applied field. The variations in an otherwise homogenous field caused by the presence of the object is called a magnetic anomaly, and observations of such anomalies can be used to infer the presence of magnetic objects.

Because there are numerous factors that affect magnetic fields there is no unique interpretation of a set of magnetometry data. Conversely, there is no unique magnetic anomaly produced by a particular kind of buried object. Factors that influence the response of a magnetometer to buried objects include the size, shape, depth, orientation, and magnetic susceptibility of the buried material. Various magnetometers are available such that many objects of interest at hazardous waste sites (particularly buried ferromagnetic materials such as drums, tanks, pipes and iron scrap) are detectable. While the location of ferromagnetic material can be detected to the precision of the survey, difficulties may be encountered in interpreting and attempting to identify the source of magnetic anomalies.

##### **5.1.1.2 Metal Detection**

When a radio frequency electromagnetic field generated by a transmitter coil encounters a highly conductive object such as metal (not necessarily ferromagnetic), alternating currents are induced in the object that, in turn, generate alternating secondary magnetic fields that are detected as alternating voltages by a receiver coil. The presence of the metal object effectively "couples" the transmitter and receiver coils, which otherwise are oriented so that little or no coupling exists. The principles of metal detector operation are very similar to those associated with electromagnetic induction instruments.

A number of factors influence the response of a metal detector. The receiver response increases with the size and surface area, and decreases with the depth of a buried object. Factors such as soil properties and object shape complicate detectability and interpretation. Certain shapes, such as elongated metal rods, are difficult to detect. Iron minerals and conductive fluids will affect the detector response in much the same manner as a target of interest. Generally, metal detectors show greater response to smaller nearby targets than to larger targets at greater depth, and the presence of widespread metallic debris at a site can interfere with attempts to detect buried drums and other objects.

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## **5.1.2 General Applicability**

### **5.1.2.1 Magnetic Surveys (Magnetometry)**

Magnetometer and gradiometer surveys are useful in environmental and engineering projects that require a technique for mapping buried vertically oriented metallic pipe (e.g., locating a buried water well casing), or mapping stratigraphy or geologic structure in an igneous settings. Magnetometers are also a useful tool for mapping buried metallic debris, metallic utilities or metallic containers.

Magnetic surveys can more readily detect metallic masses than identify or characterize targets. Calculations of the mass or size of detected objects generally yield only approximate results.

Magnetic surveys may be impractical in areas where metal pipes, fences, railroad tracks, metal buildings, and other ferrous metal artifacts are abundant. However, proper selection of equipment and survey techniques can often alleviate some of these problems.

### **5.1.2.2 Metal Detectors**

Metal detectors (MDs) can be used for locating buried metallic containers of various sizes; defining the boundaries of trenches containing metallic containers; locating buried metallic storage tanks; locating buried metallic pipes; avoiding buried utilities when drilling or trenching; or locating utility trenches which may provide a permeable pathway for contaminants.

The detection range of a MD is relatively short. Its sensitive areas are focused directly above and below the coil providing good definition of object location. Quart-sized metal objects can be detected at a distance of about 1 meter; objects the size of a 55 gallon drum can be detected up to 3 meters; and massive piles of metals can be detected at depths of 3 to 6 meters. Deeper objects are difficult to detect with an MD. Although most MDs are operated on foot, some can be vehicle-mounted if desired.

## **5.1.3 Instrumentation**

### **5.1.3.1 Magnetometers**

Three types of magnetometers, the fluxgate, proton precession, and the cesium vapor magnetometers, are commonly used at hazardous waste sites. The fluxgate magnetometer uses an iron core of high magnetic susceptibility as a sensor. The amount of coiled electrical current necessary to induce magnetic saturation of the rod is directly dependent upon, and thus measures, the strength of the ambient magnetic field. In a proton precession magnetometer a strong magnetic field is applied to a sensor filled with proton-rich fluid (e.g., kerosene) that realigns the protons. The field is then turned off and the frequency of the signal generated by the protons as they realign themselves ("precess") to the earth's magnetic field is dependent upon and measures the strength of the field at that point. The third common type of magnetometer is the cesium vapor (alkali-vapor) magnetometer. The cesium vapor magnetometer is capable of obtaining an order of magnitude greater sensitivity than the proton precession magnetometer. The cesium vapor magnetometer operates via a beam of polarized light from a cesium vapor lamp that is passed through a cell of cesium vapor. The atoms of the vapor become excited as they absorb greater amounts of the polarized light. The vapor in the cell eventually reaches an energy state that can no longer absorb the light and renders the cell transparent. A radio-frequency magnetic field causes the atoms of the vapor to shift back to an energy state that allows the vapor to again absorb the polarized light. The frequency required to return the vapor to an energy state that allows the cell to absorb light is a function of the ambient magnetic field. Some magnetometers, such as the fluxgate, are extremely

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sensitive to orientation during measurement. In order to alleviate this problem, two sensors are connected by a rigid pole to form a "gradiometer" that measures only a certain directional component of the earth's magnetic field. These gradiometers are commonly used at hazardous waste sites.

The type of magnetometer best suited for a particular site investigation depends upon characteristics of that site and should be chosen by a person familiar with the different instruments available. Proton precession magnetometers, while very useful in many situations, will cease to function in an area with high magnetic gradients such as a junkyard or near a steel bridge.

Different instruments have different levels of sensitivity. Whereas in some cases, high sensitivity may be desired to detect deeply buried objects, in other instances, a low sensitivity instrument may be desired to reduce the effects of "noise" from nearby fences or cars. Furthermore, the size of the survey area and the resolution required will determine whether the magnetometer used is hand-held for stationary measurements or a vehicle-mounted continuous sensor model.

#### 5.1.3.2 Metal Detectors (MDs)

Three general classes of metal detectors are commonly used in hazardous waste site studies: pipeline/cable locators, conventional "treasure hunter" detectors, and specialized detectors. The pipeline/cable detectors are commonly used by EPA field investigation teams. They do not respond to small objects like soda cans. Although most of the "treasure hunter" type detectors are used for locating coin-sized objects, some can be fitted with larger sensor coils suitable for detection of larger objects at greater depths. Some of these models also can operate under adverse soil conditions such as soils high iron content. Specialized detectors are also available to operate to greater depths, over a wide sweep area, operate continuously, cope with special field problems, or operate while vehicle-mounted. These special MDs require an experienced operator and are not commonly available.

### 5.2 Data Acquisition

#### 5.2.1 Field Procedures

##### 5.2.1.1 Magnetics

Magnetic measurements are generally made in a cross-grid pattern, or if a continuous sensor is used, in a series of parallel lines across the survey area. The desired resolution (reconnaissance or high density) and the size and depth of the objects sought, determines the spacing of measurement stations or survey lines. Because of the phenomenon of temporal magnetic drift, a magnetic survey must include a base station where magnetic measurements are made at regular intervals. A separate base station magnetometer is used to monitor fluctuations in the earth's magnetic field. The base station magnetometer is time synchronized with the mobile magnetometer and placed in an area of the site believed to be free of metals or other anthropogenic features. The base station magnetometer is configured to record one data point at a set time interval (e.g., every 5 seconds). At the completion of the survey, the base station magnetometer is interfaced with the mobile magnetometer and the total field magnetic data are automatically corrected for any observed diurnal drift.

Special care must be taken with handling of the magnetometer during use. The operator must not take measurements with the sensor near ferromagnetic objects such as belt buckles or steel-toed boots. The orientation of the magnetometer and its height from the ground must also be carefully controlled during operation. Recorded data must be annotated with station locations to allow construction of a site magnetic map.

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### 5.2.1.2 Metal Detector

Surveys with metal detectors are similar in planning to those with magnetometers. A grid pattern of evenly spaced parallel lines is used. Desired resolution and the size of objects determine spacing. In some cases, elevating the MD a few feet off the ground may help to eliminate noise from small surface objects. An experienced operator is recommended. Recorded data must be annotated with station locations to allow construction of a site metal detection map.

## 5.2.2 **Data Format**

### 5.2.2.1 Magnetics

Most magnetometers are equipped with a solid state data logger that records the total field magnetic and/or the vertical gradient values, the survey line location, the survey station location and the time of the reading. The common units (SI) for total field magnetic data are Teslas (T). Magnetometers record total field data in units of nanoTeslas (nT). However, older texts may also refer to magnetic values as gammas (one gamma equals one nanoTesla). Vertical gradient data are commonly recorded in units of nanoTeslas per meter (nT/M).

### 5.2.2.2 Metal Detection

The data provided by a metal detector is less quantitative than that of a magnetometer. The MD signal strength may vary (depending on the instrument) with object depth, size, and shape, but this signal does not translate into a quantity such as field strength. It merely indicates the presence of a metal object. This on/off type of signal is useful because it can indicate the boundaries of a metal-bearing zone more clearly than some quantitative data such as magnetometer recordings.

## 5.3 **Data Interpretation**

### 5.3.1 **Magnetics**

#### 5.3.1.1 Correction of Diurnal Variations

Diurnal drift is automatically corrected for by interfacing the mobile magnetometer with the base station magnetometer. However, all of the diurnal corrections should be checked to verify the values and insure against instrument malfunction.

#### 5.3.1.2 Depth Estimates from Total Field

The width of a magnetic anomaly is proportional to the depth (or distance) of the source from the magnetometer sensor; the deeper the source, the broader the anomaly. This relationship is of primary importance in interpreting the results of a magnetic survey. The proportion between the width of an anomaly and the depth of the source is a function of the fall-off rate, or the variation of anomaly amplitude with distance(d). For a dipole, the total-field anomaly amplitude varies as  $1/d^3$ , and for a monopole as  $1/d^2$ . In actual practice, source orientation and other factors may result in fall-off rates from  $1/d$  to  $1/d^3$ . The shape of the magnetic profile of an anomaly and knowledge of the source object help in selecting the proper fall-off rate for depth estimation. A range of depths determined from several fall-off rates may be the most appropriate way to present depth estimates.

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In general the anomaly width is on the order of one to three times the depth of the source. Thus, for an anomaly with a width of 100 feet, the source is probably between 30 and 100 feet deep (or distant). Several methods, including the half-width rule and the slope technique, can be used to estimate source depths from total field profiles.

#### 5.3.1.3 Half-Width Rule

The half-width ( $x_{1/2}$ ) of an anomaly on a total field profile is the horizontal distance between the principal maximum (or minimum) of the anomaly (assumed to be over the center of the source) and the point where the total field value is exactly one-half of the principal maximum (Figure 3.2-1). A profile that is used for depth estimation by using the half-width rule should be oriented perpendicular to the long axis of the anomaly to give the narrowest profile. This rule is valid only for forms such as spheres, cylinders, and other simple shapes. For example, a single upright 55-gallon steel drum can be approximated as a vertical cylinder (monopole) and the depth ( $d$ ) =  $1.3 x_{1/2}$ . A buried trench filled with drums can be approximated by a horizontal cylinder, where  $d = 2 x_{1/2}$ .

#### 5.3.1.4 Slope Techniques

Depth of the source can be estimated using the slope of the anomaly at the inflection points of the profile. The horizontal extent ( $X_z$ ) of the "straight" portion of the slope is determined as shown in Figure 3.2-1. The depth is then estimated by the equation,

$$d = KX_z \text{ where } 0.5 < K < 1.5$$

### 5.3.2 **Metal Detection**

Very little interpretation is necessary for metal detection surveys performed to provide qualitative data on the presence of metallic objects in the survey area, as a precursor to more detailed subsequent geophysical surveys. For these cases, the positive audible responses or meter deflections are recorded on site grid maps and no further processing or interpretation is made. More detailed metal detection surveys using strip-chart or magnetic tape recording are possible. Typically, data are plotted on site grid maps following computer processing. Corrections for nonlinearities and smoothing of the data to eliminate small-target responses can be accomplished.

### 5.4 Applications Management

#### 5.4.1 **Prerequisites**

As described in Section 5.1.2, appropriate planning of magnetic and metal detection surveys requires at least a basic understanding of general site features and hydrogeologic characteristics, as well as the probable variability in conditions. The Work Plan should describe, in as much detail as possible, the known site conditions which may affect the measurements, and the objectives of proposed survey efforts. The type and degree of data interpretation and the desired format for data presentation should be specified if possible.

#### 5.4.2 **Work Planning and Scheduling**

Magnetic and metal detection surveys may be performed concurrently with field investigations, in which case on-site interpretation of data may provide real-time guidance for well drilling activities. Ideally,

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however, these geophysical surveys should be conducted in advance, allowing sufficient time for data interpretation and use of the results in planning other field exercises.

The time and effort required by these geophysical surveys vary greatly depending on the site-specific objectives and site conditions. Typically, 2 to 10 acres of metal detection can be performed by one person per day, depending on the complexity of the site. Additionally, 2 to 3 linear miles of magnetometer data can be collected by 1 person per day. Data reduction and interpretation will require at least an equivalent amount of time to the field work. Weather conditions, terrain, and obstructive site features cause considerable variability in these estimates.

## **5.5            Calibration**

### **5.5.1         Magnetic Survey**

Magnetometer readings should be compared regularly to readings of a reference base station magnetometer; this procedure is necessary if corrections are to be made for changes in the earth's magnetic field over time.

#### **5.5.1.1      Daily Quality Control**

All data sets should be accompanied by quality control data that indicates the level of quality of each individual data point. Periodically, replicate measurements should be made so that measurement precision can be established. This procedure also requires corrections for variations in the earth's magnetic field with time. Each data set should be referenced to the most recent calibrations. All data obtained prior to a calibration requiring significant changes in instrument controls are suspect, and the measurements should be repeated or otherwise validated. Data should be preliminarily reduced and plotted during the field program to determine the overall quality of the data and whether the survey results are consistent with the site conceptualization. Data points representing discontinuities in the curves should be validated by repetition and, if necessary, a fine grid of measurements made to determine whether the anomaly represents a site feature of interest, a spurious reading, or an obstructive interference.

The earth's magnetic field varies constantly due, primarily, to solar activity. These natural fluctuations must be accounted for and removed from the survey data. A second magnetometer will be used as a base station to measure and record these fluctuations. These data will subsequently be used to drift correct the survey magnetometer data. In addition, the U.S. Space Environmental Agency should be contacted daily to obtain the latest solar activity forecasts. Data acquisition will cease in the event of a magnetic storm. The phone number for the U.S. Space Environmental Agency is (303) 497-3171.

### **5.5.2         Metal Detection**

#### **5.5.2.1      Calibration**

Metal detectors normally are not calibrated, and only relative response is of interest. Periodically, the sensitivity should be checked by nulling the instrument at a fixed location known to be free of metal, and adjusting the gain to provide a proper response over a known target.

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#### 5.5.2.2 Daily Quality Control

Metal detector data should be accompanied by sufficient quality control data to verify that the instrument was operating properly. Occasional repetitive measurements and a log of the sensitivity adjustments usually suffice for this purpose.

### 5.6 LIMITATIONS

Magnetometer data may be adversely affected by the presence of anthropogenic surface features such as buildings, fences, power lines, vehicles, reinforced concrete, and other metal objects. The magnetic response from these surface features can be much larger than that due to a single buried steel drum, and thus can mask the response of a drum. Magnetometer surveys should not be conducted in urban areas or areas where surface anthropogenic features are prevalent.

Data interpretation is not always straightforward with magnetic data. Metallic drums that are buried at the same depth but at different orientations can yield very dissimilar instrument responses. Data that are collected along survey lines oriented east-west can appear very different than data that are collected along survey lines oriented north-south (the preferred survey orientation).

### 6.0 REFERENCES

Good discussions of various geophysical survey techniques and applications are found in the following references:

Benson, R. C., R. A. Glaccum and M. R. Noel, 1982. Geophysical Techniques for Sensing Buried Wastes and Waste Migration, Technos, Inc., Miami, Florida, Contract No. 68-03-3050, U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

Costello, R. L., 1980. Identification and Description of Geophysical Techniques, Report No. DRXTH-TE-CR-80084, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland; Defense Technical Information System Number ADA 123939.

McKown, G. L., G. A. Sandness and G. W. Dawson, 1980. Detection and Identification of Buried Waste and Munitions, Proceedings of the 11th American Defense Preparedness Association Environmental Systems Symposium, Arlington, Virginia, 1980.

Ward, Stanley, H. 1990. Geotechnical and Environmental Geophysics, Society of Exploration Geophysicists. Tulsa, Oklahoma.

### 7.0 RECORDS

The following information will be recorded in the field logbook.

- Date
- Equipment operators
- Name and project number of site
- Position and instrument readings or responses if not recorded by a data logger
- Position-specific information
- Field sketches



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
IN-SITU HYDRAULIC CONDUCTIVITY TESTING

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## 1.0 PURPOSE

This guideline is intended to describe procedures for performing in-situ hydraulic conductivity testing (slug testing) in boreholes and monitoring wells, and provide a short description of commonly used evaluation techniques for the data generated. Slug tests are used to provide data regarding the hydraulic properties of the formation tested. A variation of the slug test, called a constant-head test, is also briefly described.

## 2.0 SCOPE

Slug tests are short-term tests designed to provide approximate hydraulic conductivity values for the portion of a formation immediately surrounding the screened/open interval of a well or boring. These tests are much less accurate than pumping tests, as a much more localized area is involved. Therefore, a number of slug tests are typically performed and averaged to determine a representative hydraulic conductivity value for the formation tested. Performance of slug tests may be preferable to pumping tests in situations where handling of large volumes of contaminated water is a concern or when time/budget constraints preclude the more expensive and time-consuming setup and performance of a pumping test.

Constant-head tests also are used to determine hydraulic conductivity values and are similar to slug tests with regard to the quality of data obtained and time/cost considerations. A disadvantage of constant-head tests is that a significant volume of water may be added to high-permeability formations, potentially affecting short-term water quality.

## 3.0 GLOSSARY

Hydraulic Conductivity (K) - A quantitative measure of the ability of a porous material to transmit water, defined as the volume of water that will flow through a unit cross-sectional area of porous material per unit time under a head gradient of 1. Hydraulic conductivity is dependent upon properties of the medium and fluid. Common units of expression include centimeters per second (cm/sec), feet per day (ft/day), and gallons per day per foot<sup>2</sup> (gpd/ft<sup>2</sup>).

Transmissivity (T) - A quantitative measure of the ability of an aquifer to transmit water. The product of the hydraulic conductivity times the saturated thickness.

Slug Test - A rising head or falling head test used to measure hydraulic conductivity. A slug test consists of instantaneously changing the water level within a well and measuring the rate of recovery of the water level to equilibrium conditions. Slug tests are performed by either withdrawing a slug of water (rising head test) or adding a slug of water (falling head test), then measuring recovery over time. A solid slug of known volume can be used to displace a volume of water, thereby simulating the addition or removal of water.

## 4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist, in conjunction with the Project Manager, shall evaluate the type(s) and extent of hydraulic testing required for a given project during the planning process, and design the field program accordingly. The project hydrogeologist also shall ensure that field personnel have the necessary training and guidance to properly perform the tests, and shall oversee data reduction activities, including selecting the appropriate evaluation techniques and checking calculations for accuracy.

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Field Geologist - The field geologist is responsible for performing the planned field tests as specified in the project planning documents, (or approved modifications thereto). The field geologist also generally assists in the data evaluation process. The field geologist shall be knowledgeable in the testing methodologies used and is responsible for obtaining the necessary support equipment required to perform the field tests. All applicable data regarding testing procedures, equipment used, well construction, and geologic/hydrogeologic conditions shall be recorded by the field geologist. The field geologist shall be familiar enough with testing procedures/requirements to be able to recommend changes in methodology, should unanticipated field conditions be encountered.

## 5.0 PROCEDURES

### 5.1 In-situ Hydraulic Conductivity Testing in Wells

Slug tests are commonly performed in completed wells. Prior to testing, the well shall be thoroughly developed and allowed to stabilize, in order to obtain accurate results. Once the water level within the well has stabilized at its static level, it shall be quickly raised or lowered and the rate of recovery measured.

One of the basic assumptions of slug testing is that the initial change in water level is instantaneous; therefore, an effort shall be made to minimize the time involved in raising or lowering the water level initially. Various methods can be used to induce instantaneous (or nearly instantaneous) changes in water level within the well. A rise in water levels can be induced by pouring water into the well. A solid slug of known volume, quickly lowered below the water level within the well, will displace an equivalent volume of water and raise the water level within the well. The slug can be left in place until the water level restabilizes at the static water level, then suddenly removed to create a drop in water level within the well. An advantage of using a solid cylinder of known volume (slug) to change the water level is that no water is removed or added to the monitoring well. This eliminates the need to dispose of contaminated water and/or add water to the system. A bailer or pump can be used to withdraw water from the well. If a pump is used, pumping shall not continue for more than several seconds so that a cone of depression is not created which would adversely impact testing results. The pump hose shall also be removed from the well during the recovery period, as data analysis techniques involve volume of recovery versus time, and leaving the hose within the well would distort the calculated testing results by altering the apparent volume of recovery. Falling head slug tests should only be performed in wells with fully submerged screens, while rising head slug tests can be performed in wells with either partially or fully submerged screens/open intervals.

Other methods that can be used to change water levels within a well include creating a vacuum or a high pressure environment within the well. The vacuum method will raise water levels within the well, while the pressure method will depress the water level in the well. These methods are particularly useful in highly permeable formations where other methods are ineffective in creating measurable changes in water levels. Both of these methods are limited to wells which have completely submerged screens.

Rate of recovery measurements shall be obtained from time zero (maximum change in water level) until water level recovery exceeds 90 percent of the initial change in water level. In low permeability formations, the test may be cut-off short of 90 percent recovery due to time constraints. Time intervals between water level readings will vary according to the rate of recovery of the well. For a moderately fast recovering well, water level readings at 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.75, 1.0, 1.25, 1.5, 2.0, 2.5, 3.0, 4.0, . . . minutes may be required. With practice, readings at down to 0.05-minute (3 seconds) time intervals can be obtained with reasonable accuracy, using a pressure transducer and hand held readout. For wells which recover very fast, a pressure transducer and data logger may be required to obtain representative data. Time intervals between measurements can be extended for slow recovering wells. A typical

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schedule for measurements for a slow recovering well would be 0, 0.25, 0.5, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0, 8.0, 10.0, 15.0, 20.0, 30.0, . . . minutes from the beginning the test. Measurements shall be taken from the top of the well casing.

Water level measurements can be obtained using an electric water level indicator, popper, or pressure transducer. Steel tape coated with chalk or water sensitive paste although very accurate, is a slower method of obtaining water levels and is generally not recommended for use due to the frequency at which water level measurements need to be obtained during the performance of a slug test.

Time/recovery should be field-plotted on semilog graph paper to determine the data quality. The data set should plot along a sloped, straight line. If excessive data scatter is observed, the test should be rerun until acceptable results are obtained.

The following data shall be recorded when performing slug tests in wells or borings:

- Well/boring ID number
- Total depth of well/boring
- Screened/open interval depth and length
- Gravel pack interval depth and length
- Well stickup above ground surface
- Gravel pack radius
- Static water level
- Aquifer thickness
- Depth to confining layer
- Time/recovery data

A variation of the slug test, called a constant-head test, is a test in which water is added to the well at a measured rate sufficient to maintain the water level in the well at a constant height above the static water level. Once a stable elevated water level has been achieved, discharge (pumping) rate measurements are recorded in place of time/recovery data for approximately 10 to 20 minutes. The hydraulic conductivity is then calculated from this information. The constant-head test is generally not recommended for monitoring wells as large volumes of water may be introduced into the screened formation, potentially impacting later sampling events.

## **5.2 In-situ Hydraulic Conductivity Testing in Borings**

Slug tests can be performed in borings while the boring is being advanced. This permits testing of formations at different depths throughout the drilling process. Boreholes to be tested shall be drilled using casing, so that discrete depths may be investigated. Various tests and testing methods are described below. The most appropriate test and testing method to be used in a situation varies and shall be selected after a careful evaluation of drilling, geologic, and general site conditions.

Rising head or falling head slug tests can be performed in saturated and unsaturated formations during drilling. There are two ways that the tests can be performed. One way entails setting the casing flush with the bottom of the boring when the desired testing depth has been reached. The hole is then cleaned out to remove loose materials, the drill bit and rods are carefully withdrawn from the boring, and a few feet of sand (of higher permeability than the surrounding formation) is added to the bottom of the boring. After the water level in the boring has stabilized (for saturated formations), the static water level is measured and recorded. The water level is then raised (falling head test) or lowered (rising head test) and the change in water level is measured at time intervals determined by the field hydrogeologist. Only falling head tests can be performed for depth intervals within the unsaturated (vadose) zone. As described for

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wells, time intervals for water level measurements will vary according to the formation's hydraulic conductivity. The faster the rate of recovery expected, the shorter the time intervals between measurements shall be. The rate of change of water level will be used to calculate hydraulic conductivity. The test is to be conducted until the water level again stabilizes, or for a minimum of 20 minutes. In low permeability formations, it is not always practical to run the test until the water level stabilizes, as it may take a long time to do so. The top of the casing shall be used as the reference point for all water level measurements.

The second method for conducting a slug test during drilling consists of placing a temporary well with a short screen into the cleaned-out boring, pulling the drilling casing back to expose the screen, allowing the formation to collapse around the screen (or placing a sand/gravel pack around the screen), and performing the appropriate hydraulic conductivity test in the well, as described for the first method. Again, the test shall be conducted until the water level stabilizes or for a minimum of 20 minutes. This method allows for testing a larger section of the formation and results in more reliable hydraulic conductivity estimates.

Constant-head tests may also be performed in borings. As described for monitoring wells, once a stable elevated level has been achieved, the discharge rate into the boring is measured for a period of time, usually 10 to 20 minutes, and the hydraulic conductivity is calculated from this. This method is the most accurate method depicted in this section, and shall be given preference over others if the materials are available to perform the test and the addition of water to the boring does not adversely impact project objectives. Once the test is over, additional information can be gathered by measuring the rate of the drop in water level in the boring (for saturated formations). A limitation of the constant-head test is that foreign water is introduced into the formation which must be removed from the well area by natural or artificial means, before a representative groundwater sample can be obtained.

Detailed descriptions regarding the performance of borehole hydraulic conductivity tests and subsequent data analysis techniques are provided in Ground Water Manual (1981).

### 5.3 Data Analysis

There are a number of data analysis methods available to reduce and evaluate slug testing data. The determination of which method is most appropriate shall be made based on the testing conditions (including physical setup of the well/boring tested, hydrogeologic conditions, and testing methodology) and the limitations of each test analysis method. Well construction details, aquifer type (confined or unconfined), and screened/open interval (fully or partially penetrating the aquifer) shall be taken into account in selecting an analysis method. Cooper, et al. (1967), and Papadapulos, et al. (1973) have developed test interpretation procedures for fully penetrating wells in confined aquifers. Hvorslev (1951) developed a relatively simple analytical procedure for point piezometers in an infinite isotropic medium. In Cedergren (1967), Hvorslev presents a number of analytical procedures which cover a wide variety of hydrogeologic conditions, testing procedures, and well/boring/piezometer configurations. Bouwer and Rice (1976) developed an analytical technique applicable to both unconfined and confined conditions, which factors in partial/full penetration and discusses well screen gravel pack considerations. The Ground Water Manual (1981) presents a number of testing and test analysis procedures for wells and borings open above or below the water table, and for both falling head and constant-head tests. The methods described above do not represent a complete listing of test analysis methods available, but are some of the more commonly used and accepted methods. Other methods can be used, at the discretion of the project hydrogeologist and in concurrence with the Project Manager and client.

One consideration to be noted during data analysis is the determination of the screened/open interval of a tested well. If a well with a fully submerged screen is installed in a relatively low permeability formation,

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and a gravel pack which is significantly more permeable is installed around the screen, the length of the gravel pack (if longer than the screened interval) should be used as the screened/open length, rather than the screen length itself. In situations where the formation permeability is judged to be comparable to the gravel pack permeability (within about an order of magnitude) this adjustment is not required.

All data analysis applications and calculations shall be reviewed by technical personnel thoroughly familiar with testing and test analysis procedures. Upon approval of the calculations and results, the calculation sheets shall be initialed and dated by the reviewer. Distribution copies shall be supplied to appropriate project personnel and the original copy stored in the project central file.

## 6.0 REFERENCES

Cedergren, H. R., 1967. Seepage, Drainage, and Flow Nets. John Wiley and Sons Inc., New York, pp. 78-76.

Cooper, H. H., Jr., J. D. Bredehoeft, and I. S. Papadopoulos, 1967. Response of a Finite-Diameter Well to an Instantaneous Change of Water. Water Resources Research, V. 3, No. 1, pp. 263-269.

Hvorslev, M. J., 1951. Time Lag and Soil Permeability in Ground Water Observations. U.S. Army Corps of Engineers, Waterways Experiment Station, Washington, D.C., Bull. No. 36.

Papadopoulos, I. S., J. D. Bredehoeft, and H. H. Cooper, 1973. On the Analysis of Slug Test Data. Water Resources Research, V. 9, No. 4, pp. 1087-1089.

Bouwer, H. and R. C. Rice, 1976. "A Slug Test for Determining Hydraulic Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells." Water Resources Research, 12:423-28.

United States Department of the Interior, 1981. Ground Water Manual. U.S. Government Printing Office, Denver, Colorado.

## 7.0 RECORDS

Field data shall be recorded on the data sheet included as Attachment A (or equivalent). 1 Any notes regarding testing procedures, problems encountered, and general observations not included on the data sheet shall be noted in the bound site logbook or field notebook. The boring log and well construction diagrams for each well/boring tested shall be used as references during testing and data analysis activities. Original data sheets shall be placed in the project file, along with the logbook/notebook.

---

1 If an automated data recorder is used, the data may be displayed using the printer output from the unit. Such printouts should be annotated to include the relevant data form, or attached to the form shown as Attachment A.

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ATTACHMENT A  
EXAMPLE HYDRAULIC CONDUCTIVITY TESTING DATA SHEET



**HYDRAULIC CONDUCTIVITY TESTING DATA SHEET**

PROJECT NAME: ..... WELL/BORING NO.: .....

PROJECT NO.: ..... GEOLOGIST: .....

WELL DIAMETER: ..... SCREEN LENGTH/DEPTH: ..... TEST NO.: .....

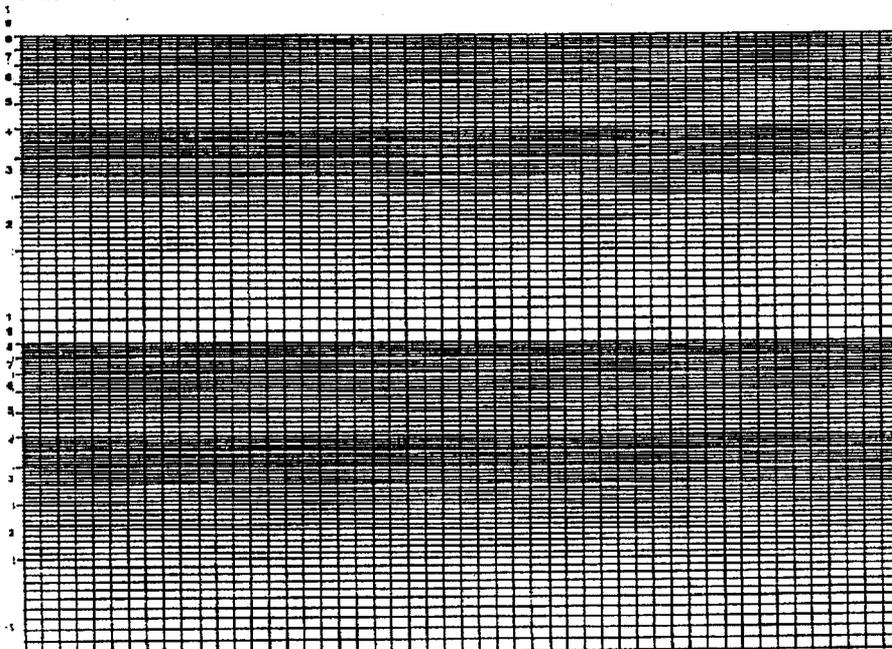
STATIC WATER LEVEL (Depth/Elevation): ..... DATE: .....

TEST TYPE (Rising/Falling/Constant Head): ..... CHECKED: .....

METHOD OF INDUCING WATER LEVEL CHANGE: ..... PAGE ..... OF .....

REFERENCE PT. FOR WL MEAS. (Top of Casing, Transducer, etc.): .....

ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD ( $\Delta H$ ) (feet)	ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD ( $\Delta H$ ) (feet)	WELL SCHEMATIC



REMARKS:

.....

.....

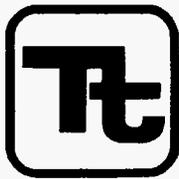
.....

.....

.....

.....

CALCS, SKETCH MAPS, ETC.:



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Health & Safety		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
UTILITY LOCATING AND EXCAVATION CLEARANCE

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## 1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

## 2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

## 3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

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#### 4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

#### 5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

##### 5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.
- 2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scars and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

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locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.
4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.
5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain
6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.
7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.
8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

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**5.2            Overhead Power Lines**

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

<u>Nominal Voltage</u>	<u>Minimum Clearance</u>
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

**6.0            UNDERGROUND LOCATING TECHNIQUES**

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

**6.1            Geophysical Methods**

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

**Electromagnetic Induction**

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

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## **Magnetics**

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

## **Ground Penetrating Radar**

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

### **6.2 Passive Detection Surveys**

#### **Acoustic Surveys**

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

#### **Thermal Imaging**

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

### **6.3 Intrusive Detection Surveys**

#### **Vacuum Excavation**

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

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debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

### Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

### Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

## 7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.
2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.
4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

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5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

**8.0 REFERENCES**

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4  
 OSHA 29 CFR 1926(b)(2)  
 OSHA 29 CFR 1926(b)(3)  
 TtNUS Utility Locating and Clearance Policy  
 TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction  
 TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys  
 TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

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**ATTACHMENT 1  
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES**



**American Public Works Association**  
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625  
Phone (816) 472-6100 • Fax (816) 472-1610  
Web www.apwa.net • E-mail apwa@apwa.net

**ONE-CALL SYSTEMS INTERNATIONAL  
CONDENSED DIRECTORY**

<b>Alabama</b> Alabama One-Call 1-800-292-8525	<b>Iowa</b> Iowa One-Call 1-800-292-8989	<b>New Jersey</b> New Jersey One Call 1-800-272-1000
<b>Alaska</b> Locate Call Center of Alaska, Inc. 1-800-478-3121	<b>Kansas</b> Kansas One-Call System, Inc. 1-800-344-7233	<b>New Mexico</b> New Mexico One Call System, Inc. 1-800-321-2537 Las Cruces- Dona Ana Blue Stakes 1-888-526-0400
<b>Arizona</b> Arizona Blue Stake 1-800-782-5348	<b>Kentucky</b> Kentucky Underground Protection Inc. 1-800-752-6007	<b>New York</b> Dig Safely New York 1-800-862-7962 New York City- Long Island One Call Center 1-800-272-4480
<b>Arkansas</b> Arkansas One Call System, Inc. 1-800-482-8998	<b>Louisiana</b> Louisiana One Call System, Inc. 1-800-272-3020	<b>North Carolina</b> The North Carolina One-Call Center, Inc. 1-800-632-4949
<b>California</b> Underground Service Alert North 1-800-227-2600 Underground Service Alert of Southern California 1-800-227-2600	<b>Maine</b> Dig Safe System, Inc. 1-888-344-7233	<b>North Dakota</b> North Dakota One-Call 1-800-795-0555
<b>Colorado</b> Utility Notification Center of Colorado 1-800-922-1987	<b>Maryland</b> Miss Utility 1-800-257-7777 Miss Utility of Delmarva 1-800-282-8555	<b>Ohio</b> Ohio Utilities Protection Service 1-800-362-2764 Oil & Gas Producers Underground Protect'n Svc 1-800-925-0988
<b>Connecticut</b> Call Before You Dig 1-800-922-4455	<b>Massachusetts</b> Dig Safe System, Inc. 1-888-344-7233	<b>Oklahoma</b> Call Okie 1-800-522-6543
<b>Delaware</b> Miss Utility of Delmarva 1-800-282-8555	<b>Michigan</b> Miss Dig System, Inc. 1-800-482-7171	<b>Oregon</b> Oregon Utility Notification Center/One Call Concepts 1-800-332-2344
<b>Florida</b> Sunshine State One-Call of Florida, Inc. 1-800-432-4770	<b>Minnesota</b> Gopher State One Call 1-800-252-1168	<b>Pennsylvania</b> Pennsylvania One Call System, Inc. 1-800-242-1776
<b>Georgia</b> Underground Protection Center, Inc. 1-800-282-7411	<b>Mississippi</b> Mississippi One-Call System, Inc 1-800-227-6477	<b>Rhode Island</b> Dig Safe System, Inc. 1-888-344-7233
<b>Hawaii</b> Underground Service Alert North 1-800-227-2600	<b>Missouri</b> Missouri One-Call System, Inc. 1-800-344-7483	<b>South Carolina</b> Palmetto Utility Protection Service Inc. 1-888-721-7877
<b>Idaho</b> Dig Line Inc. 1-800-342-1585 Kootenai County One-Call 1-800-428-4950 Shoshone - Benewah One-Call 1-800-398-3285	<b>Montana</b> Utilities Underground Protection Center 1-800-424-5555 Montana One Call Center 1-800-551-8344	<b>South Dakota</b> South Dakota One Call 1-800-781-7474
<b>Illinois</b> JULIE, Inc. 1-800-892-0123 Digger (Chicago Utility Alert Network) 312-744-7000	<b>Nebraska</b> Diggers Hotline of Nebraska 1-800-331-5666	<b>Tennessee</b> Tennessee One-Call System, Inc. 1-800-351-1111
<b>Indiana</b> Indiana Underground Plant Protection Service 1-800-382-5544	<b>Nevada</b> Underground Service Alert North 1-800-227-2600	
	<b>New Hampshire</b> Dig Safe System, Inc. 1-888-344-7233	

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**ATTACHMENT 1 (Continued)**

**Texas**

Texas One Call System  
1-800-245-4545  
Texas Excavation Safety System, Inc.  
1-800-344-8377  
Lone Star Notification Center  
1-800-669-8344

**Utah**

Blue Stakes of Utah  
1-800-662-4111

**Vermont**

Dig Safe System, Inc.  
1-888-344-7233

**Virginia**

Miss Utility of Virginia  
1-800-552-7001  
Miss Utility (Northern Virginia)  
1-800-257-7777

**Washington**

Utilities Underground Location Center  
1-800-424-5555  
Northwest Utility Notification Center  
1-800-553-4344  
Inland Empire Utility Coordinating  
Council  
509-456-8000

**West Virginia**

Miss Utility of West Virginia, Inc.  
1-800-245-4848

**Wisconsin**

Diggers Hotline, Inc.  
1-800-242-8511

**Wyoming**

Wyoming One-Call System, Inc.  
1-800-348-1030  
Call Before You Dig of Wyoming  
1-800-849-2476

**District of Columbia**

Miss Utility  
1-800-257-7777

**Alberta**

Alberta One-Call Corporation  
1-800-242-3447

**British Columbia**

BC One Call  
1-800-474-6886

**Ontario**

Ontario One-Call System  
1-800-400-2255

**Quebec**

Info-Excavation  
1-800-663-9228

Subject

UTILITY LOCATING AND  
EXCAVATION CLEARANCE

Number

HS-1.0

Revision

2

Page

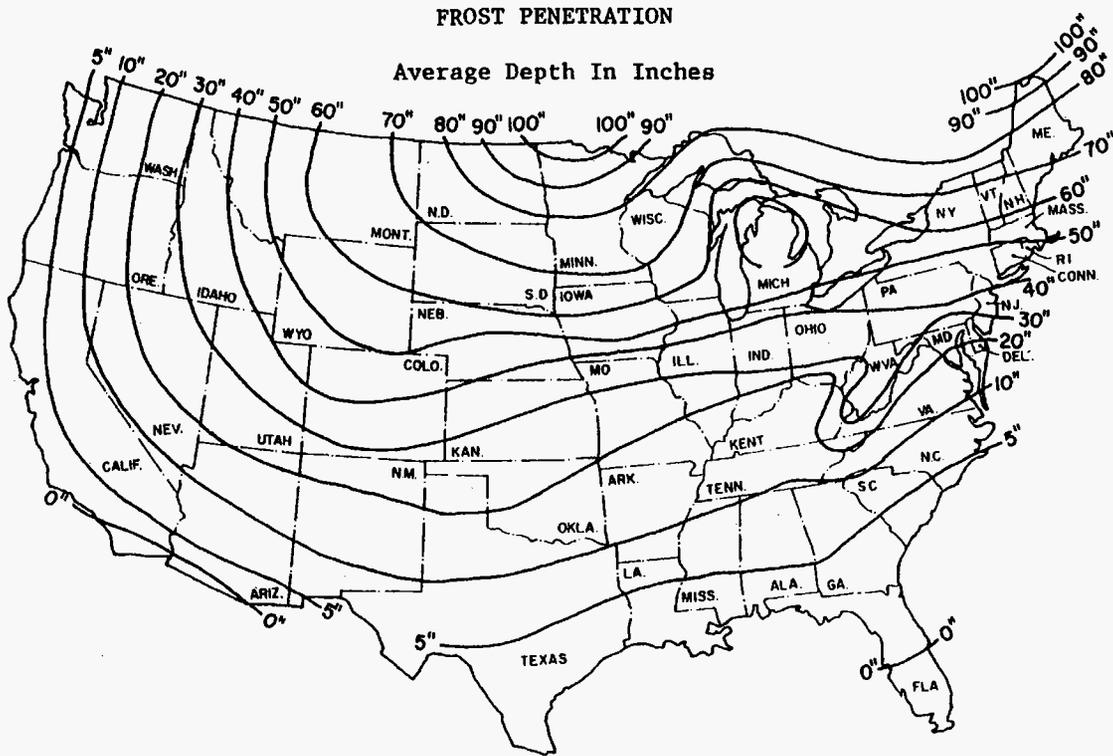
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12/03

### ATTACHMENT 2

### FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

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**ATTACHMENT 3  
UTILITY CLEARANCE FORM**

Client: \_\_\_\_\_ Project Name: \_\_\_\_\_  
 Project No.: \_\_\_\_\_ Completed By: \_\_\_\_\_  
 Location Name: \_\_\_\_\_ Work Date: \_\_\_\_\_  
 Excavation Method/Overhead Equipment: \_\_\_\_\_

1. Underground Utilities Circle One
- a) Review of existing maps? yes no N/A
  - b) Interview local personnel? yes no N/A
  - c) Site visit and inspection? yes no N/A
  - d) Excavation areas marked in the field? yes no N/A
  - e) Utilities located in the field? yes no N/A
  - f) Located utilities marked/added to site maps? yes no N/A
  - g) Client contact notified yes no N/A  
 Name \_\_\_\_\_ Telephone: \_\_\_\_\_ Date: \_\_\_\_\_
  - g) State One-Call agency called? yes no N/A  
 Caller: \_\_\_\_\_  
 Ticket Number: \_\_\_\_\_ Date: \_\_\_\_\_
  - h) Geophysical survey performed? yes no N/A  
 Survey performed by: \_\_\_\_\_  
 Method: \_\_\_\_\_ Date: \_\_\_\_\_
  - i) Hand excavation performed (with concurrent use of utility  
 detection device)? yes no N/A  
 Completed by: \_\_\_\_\_  
 Total depth: \_\_\_\_\_ feet Date: \_\_\_\_\_
  - j) Trench/excavation probed? yes no N/A  
 Probing completed by: \_\_\_\_\_  
 Depth/frequency: \_\_\_\_\_ Date: \_\_\_\_\_

2. Overhead Utilities Present Absent
- a) Determination of nominal voltage yes no N/A
  - b) Marked on site maps yes no N/A
  - c) Necessary to lockout/insulate/re-route yes no N/A
  - d) Document procedures used to lockout/insulate/re-route yes no N/A
  - e) Minimum acceptable clearance (SOP Section 5.2): \_\_\_\_\_

3. Notes:  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Approval:  
 \_\_\_\_\_  
 Site Manager/Field Operations Leader Date

c: PM/Project File  
 Program File

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**ATTACHMENT 4  
OSHA LETTER OF INTERPRETATION**

Mr. Joseph Caldwell  
Consultant  
Governmental Liaison  
Pipeline Safety Regulations  
211 Wilson Boulevard  
Suite 700  
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

***Question:** Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

*Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?*

**Answer**

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651 (Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours \* \* \* or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

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#### ATTACHMENT 4 (Continued)

When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, “acceptable means” must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either “other acceptable means” or “safe and acceptable means.” The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified “careful probing or hand digging” as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language “to allow other, *equally effective means* of locating such installations.” The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used where the utilities have not been marked and detection equipment is not being used – “probing with hand-held tools.” This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

OSHA received two comments \* \* \* and input from ACCSH [OSHA’s Advisory Committee on Construction Safety and Health] \* \* \* on this provision. All commenters recommended dropping ‘such as probing with hand-held tools’ from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of “acceptable means” in the final provision.

#### Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a “shooter” (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an “acceptable means” for locating underground utilities.

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**ATTACHMENT 4 (Continued)**

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director  
Directorate of Construction

**NOTE:** OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA's interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at <http://www.osha.gov>.



TETRA TECH

# STANDARD OPERATING PROCEDURES

Number	SA-1.1	Page	1 of 34
Effective Date	04/07/2008	Revision	7
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject  
GROUNDWATER SAMPLE ACQUISITION AND  
ONSITE WATER QUALITY TESTING

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## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

## 2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

## 3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

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Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol (<sup>0</sup>/<sub>00</sub>) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

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- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

## 5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

**Knee injuries** – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

**Slips, Trips, and Falls** – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

**Cuts and Lacerations** – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.

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- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

**Vehicular and Foot Traffic Hazards** – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- **Face Traffic.** Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

## 6.0 PROCEDURES

### 6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

#### **CAUTION**

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

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Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

**CAUTION**

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.
2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.
3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

**CAUTION**

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:
  - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.
  - While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

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- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

## 6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
  - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
  - pH Paper
  - Camera and film (if appropriate)
  - Appropriate keys (for locked wells)
  - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.
- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

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### 6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons  $V = (0.163)(T)(r^2)$

where: V = Static volume of well in gallons.  
T = Linear feet of water in the well.  
r = Inside radius of well casing in inches.  
0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

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## **6.4            Evacuation of Static Water – Purging**

### **6.4.1            General**

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

### **6.4.2            Evacuation Devices**

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

#### **Bailers**

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

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Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

#### Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

#### Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

#### Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.

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- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

#### Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

#### Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

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To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

#### Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

#### 6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters

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used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

### 6.5.1 Measurement of pH

#### 6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

#### 6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

#### 6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)

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- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

#### 6.5.1.4 Measurement Techniques for Field Determination of pH

##### pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

##### pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

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2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

### **6.5.2 Measurement of Specific Conductance**

#### **6.5.2.1 General**

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

#### **6.5.2.2 Principles of Equipment Operation**

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

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### 6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

### 6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

## 6.5.3 Measurement of Temperature

### 6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

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### 6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

### 6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.
2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.
2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.
3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

## 6.5.4 Measurement of Dissolved Oxygen

### 6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

### 6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

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the two metals, reduction of oxygen to hydroxide ion (OH<sup>-</sup>) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

#### 6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

#### 6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

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7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.
8. Rinse the probe with deionized water.
9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

### **6.5.5 Measurement of Oxidation-Reduction Potential**

#### **6.5.5.1 General**

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

#### **6.5.5.2 Principles of Equipment Operation**

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

#### **6.5.5.3 Equipment**

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential**

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

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2. Thoroughly rinse the electrode with deionized water.
3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

### **6.5.6 Measurement of Salinity**

#### **6.5.6.1 General**

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

#### **6.5.6.2 Principles of Equipment Operation**

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

#### **6.5.6.3 Equipment**

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.6.4 Measurement Techniques for Salinity**

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the meter before going into the field.

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3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.
5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the probes with deionized water.

### **6.5.7 Measurement of Turbidity**

#### **6.5.7.1 General**

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

#### **6.5.7.2 Principles of Equipment Operation**

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

#### **6.5.7.3 Equipment**

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

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#### 6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

## 6.6 Sampling

### 6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

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Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

#### 6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:
  - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
  - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
  - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

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during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.
4. Calculate volume of well water to be removed as described in Section 6.3.
5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.
6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.
7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.
8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.
10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.
11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.
12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

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occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.
14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.
15. Process sample containers as described in SOP SA-6.1.
16. Decontaminate equipment as described in SOP SA-7.1.

## **6.7 Low-Flow Purging and Sampling**

### **6.7.1 Scope and Application**

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

### **6.7.2 Equipment**

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

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- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

### 6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).
2. Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.
3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.
4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well
5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

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6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.
  7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
  8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.
  9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.
  10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.
  11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:
    - pH  $\pm 0.2$  standard units
    - Specific conductance  $\pm 10\%$
    - Temperature  $\pm 10\%$
    - Turbidity less than 10 NTUs
    - DO  $\pm 10\%$
  12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.
- NOTE:** VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.
13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

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- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.
- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.
- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.
- Prepare samples for shipping as per SOP SA-6.1.

## 7.0 REFERENCES

American Public Health Association, 1989. Standard Methods for the Examination of Water and Wastewater, 17th Edition, APHA, Washington, D.C.

Barcelona, M. J., J. P. Gibb and R. A. Miller, 1983. A Guide to the Selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

Johnson Division, UOP, Inc. 1975. Ground Water and Wells, A Reference Book for the Water Well Industry. Johnson Division, UOP, Inc., Saint Paul, Minnesota.

Nielsen, D. M. and G. L. Yeates, 1985. A Comparison of Sampling Mechanisms Available for Small-Diameter Ground Water Monitoring Wells. Ground Water Monitoring Review 5:83-98.

Scaif, M. R., J. F. McNabb, W. J. Dunlap, R. L. Crosby and J. Fryberger, 1981. Manual of Ground Water Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. EPA, 1980. Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. Office of Solid Waste, United States Environmental Protection Agency, Washington, D.C.

U.S. EPA, 1994. Groundwater Sampling Procedure - Low Flow Purge and Sampling (Draft Final). U.S. Environmental Protection Agency, Region I.

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

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**ATTACHMENT A**  
**PURGING EQUIPMENT SELECTION**

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

**ATTACHMENT A**  
**PURGING EQUIPMENT SELECTION**  
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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/L length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	\$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	\$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefze®	0-100	0-500 mL/min depending on lift	\$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	\$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	\$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	\$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	\$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	\$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	\$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	\$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	\$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	\$300-400	Requires compressed gas; piezometric level indicator; other materials available.

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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	\$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	\$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	\$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	\$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	\$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	\$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	\$800-1,000	Compressed gas required; DC control module; custom built.

## Construction Material Abbreviations:

PE Polyethylene  
 PP Polypropylene  
 PVC Polyvinyl chloride  
 SS Stainless steel  
 PC Polycarbonate  
 EPDM Ethylene-propylene diene (synthetic rubber)

## Other Abbreviations:

NA Not applicable  
 AC Alternating current  
 DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

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# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T. E. Johnston</i>		

Subject  
SURFACE WATER AND SEDIMENT SAMPLING

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## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

## 2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

## 3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

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custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

## 5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

**Bridge/Boat Sampling** – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

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Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United States Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.
- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

**Entering Water to Collect Samples** – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.
- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.
- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.
- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.
- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.
- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.
- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

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Working in these areas, also recognize the following hazards and means of protection against them:

**Insects** are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellent. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

**Safety Reminder**

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

**Poisonous Plants** – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

**Temperature-Related Stress** – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

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- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.
- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder*).

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

## 6.0 PROCEDURES

### 6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

#### 6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.

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4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

**Safety Reminder**

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
- Required decontamination equipment.
- Required sample containers.
- Sealable polyethylene bags (e.g., Ziploc<sup>®</sup> baggies).
- Heavy-duty cooler.
- Ice.

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- Paper towels and garbage bags.
- Chain-of-custody records and custody seals.

#### Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.
2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.
3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.
4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.
5. Use a clean paper towel to clean and dry the outside of the container.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

#### **CAUTION**

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

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### Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.
3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
4. Raise the sampler and cap the bottle.
5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

### Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.
2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.
3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).
4. Use a paper towel to clean and dry the outside of the container.
5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

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### 6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.
- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.
- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.
- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.
- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

### 6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

### 6.3 Sediment Sampling

#### 6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

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respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

### 6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

#### **SAFETY REMINDER**

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices
- A minimum of three people are required if any of the following conditions are anticipated or observed:
  - Work in a waterway that is turbulent or swift that could sweep a sampler down stream should he or she fall in accidentally.
  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.
  - Waterway is tidal, and conditions such as those listed above could rapidly change.

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The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc<sup>®</sup> baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

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### Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.
2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

### Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.
2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.
3. Open the sampler to retrieve the sediment.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.

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6. Use a paper towel to clean and dry the outside of each container.
7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

**SAFETY REMINDER**

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.
2. Cap the top of the tube to provide suction and reduce the chance of losing the sample.
3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.
6. Use a paper towel to clean and dry the outside of each container.
7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

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All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

## 7.0 REFERENCES

American Public Health Association, 19.99 Standard Methods for the Examination of Water and Wastewater, 20th Edition, APHA, Washington, D.C.

Feltz, H. R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., V. 1, p. 271-287.

Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1984. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-84-017.

U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.



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**ATTACHMENT B  
SOIL & SEDIMENT SAMPLE LOG SHEET**



Tetra Tech NUS, Inc.

**SOIL & SEDIMENT SAMPLE LOG SHEET**

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Project Site Name: _____	Sample ID No.: _____
Project No.: _____	Sample Location: _____
<input type="checkbox"/> Surface Soil	Sampled By: _____
<input type="checkbox"/> Subsurface Soil	C.O.C. No.: _____
<input type="checkbox"/> Sediment	Type of Sample:
<input type="checkbox"/> Other: _____	<input type="checkbox"/> Low Concentration
<input type="checkbox"/> QA Sample Type: _____	<input type="checkbox"/> High Concentration

GRAB SAMPLE DATA:			
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time: _____			
Method: _____			
Monitor Reading (ppm): _____			

COMPOSITE SAMPLE DATA:				
Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method: _____				
Monitor Readings (Range in ppm): _____				

SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

<b>OBSERVATIONS / NOTES:</b>	<b>MAP:</b>

<b>Circle if Applicable:</b>	<b>Signature(s):</b>
<input type="checkbox"/> MS/MSD <input type="checkbox"/> Duplicate ID No.: _____	

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**APPENDIX C  
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION**

**C.1 Defining the Sampling Program**

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

**C.1.1 Sampling Program Objectives**

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

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### **C.1.2 Location of Sampling Stations**

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

### **C.1.3 Frequency of Sampling**

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

## **C.2 Surface Water Sample Collection**

### **C.2.1 Streams, Rivers, Outfalls and Drainage Features**

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

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Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

### **C.2.2 Lakes, Ponds and Reservoirs**

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

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### C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.
- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.
- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.



# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject  
SOIL SAMPLING

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## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

## 2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

## 3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

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Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

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- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

## 5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

**Knee injuries** – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

**Slips, Trips, and Falls** – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

**Cuts and Lacerations** - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

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- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

**Vehicular and Foot Traffic Hazards** – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

## 6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

**CAUTION**

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket.**

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## 6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

## 6.2 Soil Sample Collection

### 6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

#### 6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

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obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.
2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.
3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.
4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.
5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.
6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.
7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.
8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.
9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.
10. Label the bag with appropriate information in accordance with SOP SA-6.3.
11. Place the full sampler inside a lined cooler with ice and cool to 4°C ± 2 °C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.
12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.
13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™ samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

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#### 6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

**Safety Reminder**

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

#### **Methanol Preservation (High to Medium Level):**

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

**CAUTION**

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.
2. Pull the plunger back and insert the syringe into the soil to be sampled.
3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.
4. Weigh the sample and adjust until obtaining the required amount of sample.

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5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.
6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.
7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.
8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.
9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

**Sodium Bisulfate Preservation (Low Level):**

**CAUTION**

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.
2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation
3. Add the weighed sample to the sample vial.
4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.
5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

**NOTE**

If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

**6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses**

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

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1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.
2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.
3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.
4. Transfer the mixed soil to the appropriate sample containers and close the containers.
5. Label the sample containers in accordance with SOP SA-6.3.
6. Place the containers in a cooler of ice as soon after collection as possible.
7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

**NOTE**

Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4° C.

**NOTE**

Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

**6.2.3 Procedure for Collecting Undisturbed Soil Samples**

**NOTE**

Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:
  - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

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**REMEMBER**

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
  - Review the Safe Work Permit prior to conducting the activity.
  - Review the activity to be conducted.
2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

**CAUTION**

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.
4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.
5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.
6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.
7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.
8. Remove disturbed material in the upper end of the tube and measure the length of sample again.
9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

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10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.
11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.
12. Mark the "up" direction on the side and upper end of the tube with indelible ink.
13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).
14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

**CAUTION**

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

**CAUTION**

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

**Electrical Heating**

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

**Open Flame**

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.
- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

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### 6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

**NOTE**

Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.

For the purposes of instruction, the terms “surface soil” and “near-surface soil” are used in this SOP as follows:

- Surface soil - 0 to 6 inches bgs
- Near-surface soil - 6 to 18 inches bgs

If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon’s or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

**Safety Reminder**

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

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- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

#### **6.4 Near-Surface Soil Sampling**

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.
2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

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3. Follow steps 1 through 9 of Section 6.3.

### 6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

#### **CAUTION**

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be preceded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

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5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.
6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.
7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.
8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.
9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.
10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.
11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.
13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.
14. Follow steps 4 through 7 listed in Section 6.3.

#### 6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

#### **SAFETY REMINDER**

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such as the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

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- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

#### 6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

##### **Safety Reminder**

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

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2. Lower the sampler into the borehole inside the hollow stem auger bits.
3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.
4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.
5. Detach the sampler from the drill rods.
6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

**CAUTION**

Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.
8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.
9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings were encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).
10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.
11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).
12. Follow steps 4 through 7 in Section 6.3.

**6.7 Subsurface Soil Sampling Using Direct-Push Technology**

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

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## 6.8 Excavation and Sampling of Test Pits and Trenches

### 6.8.1 **Applicability**

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

#### **CAUTION**

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

### 6.8.2 **Test Pit and Trench Excavation**

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration

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- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example,

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samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

### **6.8.3 Sampling in Test Pits and Trenches**

#### **6.8.3.1 General**

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

#### **6.8.3.2 Sampling Equipment**

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

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- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

#### 6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

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- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:
  - a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.
  - b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.
  - c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.
  - d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.
  - e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.
  - f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.
- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

**CAUTION**

Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:
  - a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.
  - b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.
  - c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.
- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

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#### 6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable through soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

#### 6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e.g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

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Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

#### **6.8.4 Backfilling of Trenches and Test Pits**

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

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If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

## **6.9        Records**

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O<sub>2</sub> meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

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- Soil type classification

## 7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

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**ATTACHMENT A  
SOIL & SEDIMENT SAMPLE LOG SHEET**



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**SOIL & SEDIMENT SAMPLE LOG SHEET**

Page \_\_\_ of \_\_\_

Project Site Name: _____	Sample ID No.: _____
Project No.: _____	Sample Location: _____
<input type="checkbox"/> Surface Soil	Sampled By: _____
<input type="checkbox"/> Subsurface Soil	C.O.C. No.: _____
<input type="checkbox"/> Sediment	Type of Sample:
<input type="checkbox"/> Other: _____	<input type="checkbox"/> Low Concentration
<input type="checkbox"/> QA Sample Type: _____	<input type="checkbox"/> High Concentration

GRAB SAMPLE DATA:			
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time: _____			
Method: _____			
Monitor Reading (ppm): _____			

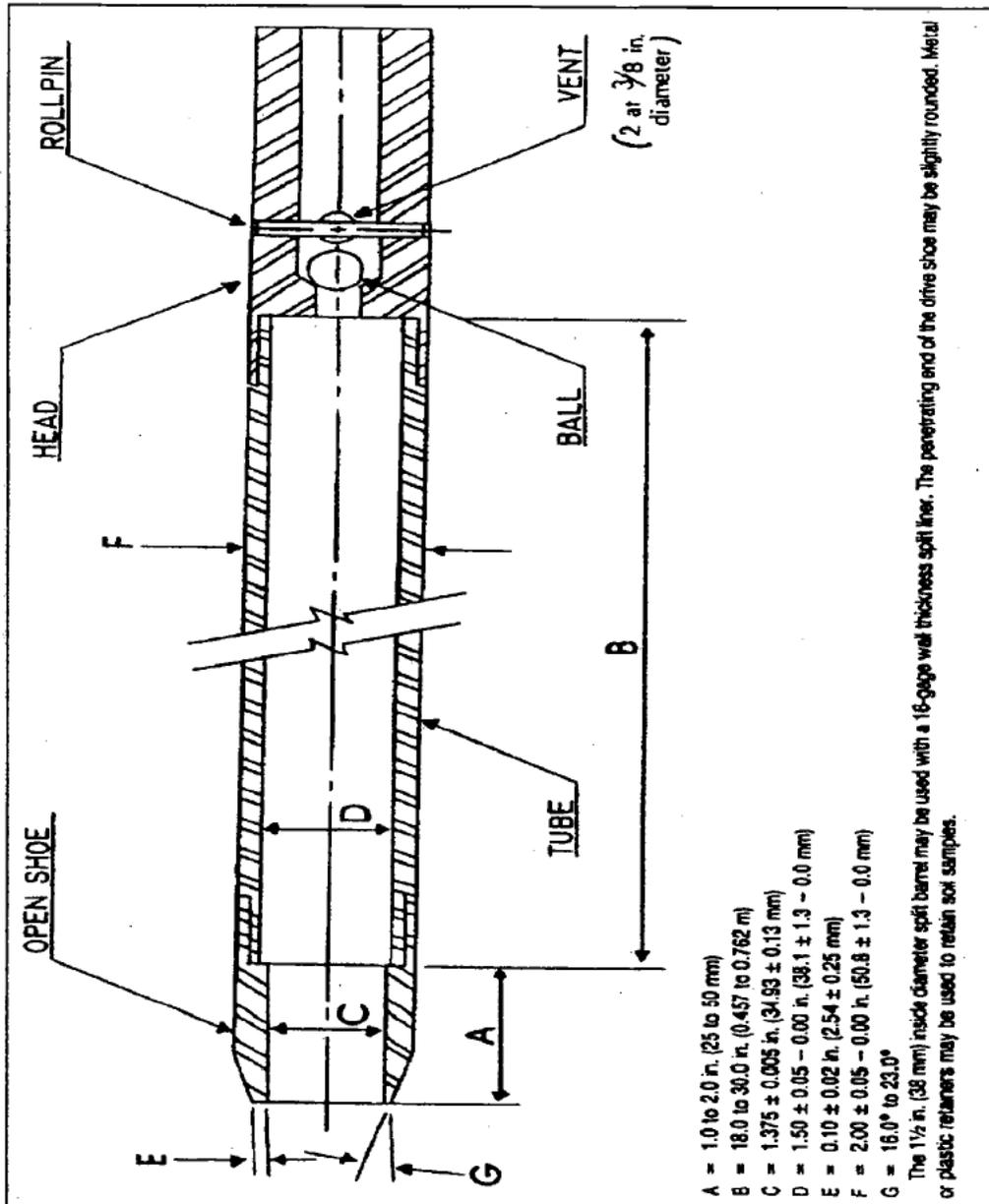
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

<b>OBSERVATIONS / NOTES:</b>	<b>MAP:</b>

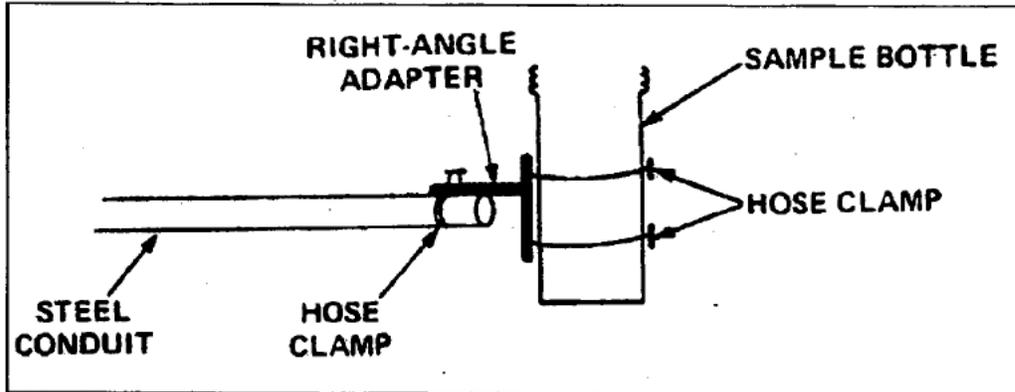
<b>Circle if Applicable:</b>	<b>Signature(s):</b>
MS/MSD      Duplicate ID No.:	

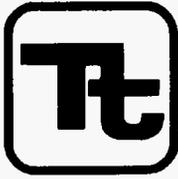
### ATTACHMENT B SPLIT-SPOON SAMPLER





**ATTACHMENT D  
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING**





TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>ds</i>		

Subject  
NATURAL ATTENUATION PARAMETER COLLECTION

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## 1.0 PURPOSE

The purpose of this document is to provide general reference information regarding natural attenuation parameter and methodology selection, sample collection, and a general understanding of the sample results.

## 2.0 SCOPE

This document provides information on selection of appropriate groundwater natural attenuation parameters, selection of sampling methods for these parameters, techniques for onsite field analysis of select parameters, and some basic understanding of the field sample results. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling practices and techniques. To a limited extent, it shall also facilitate the understanding and interpretation of the sampling results. It addresses field procedures for collection of data at sites with organic groundwater contaminants (e.g., chlorinated and petroleum hydrocarbons) to the extent practical. The focus of this document is on natural attenuation, not enhanced bioremediation.

The techniques described shall be followed whenever applicable, noting that site-specific conditions, project-specific objectives, local, state, and federal guidelines may be used as a basis for modification of the procedures noted herein. The intent of this document is to supplement the local, state, and federal guidance documents and manufacturer's analytical methods referenced in Section 6.0. It is not intended for this document to supersede this guidance or information. Please note that natural attenuation is a relatively dynamic science with ongoing research in the science and engineering community. It is important that data collectors and interpreters use the most recent regulatory guidance, which may be updated on a periodic basis from that noted in Section 6.

## 3.0 GLOSSARY

*Aerobe:* Bacteria that use oxygen as an electron acceptor.

*Anaerobe:* Organisms that can use electron acceptors other than molecular oxygen to support their metabolism.

*Anoxic groundwater:* Groundwater that contains oxygen in concentrations less than about 0.5 mg/L. This term is synonymous with the term anaerobic.

*Anthropogenic:* Man-made.

*Cometabolism:* The process in which a compound is fortuitously degraded by an enzyme or cofactor produced during microbial metabolism of another compound.

*Daughter product:* A compound that results directly from the biotic or abiotic degradation of another. For example, *cis*-1,2-dichloroethene (*cis*-1,2-DCE) is a common daughter product of trichloroethene (TCE).

*Diffusion:* The process whereby molecules move from a region of higher concentration to a region of lower concentration as a result of Brownian motion.

*Dispersion:* The tendency for a solute to spread from the path that it would be expected to follow under advective transport.

*Electron acceptor:* A compound capable of accepting electrons during oxidation-reduction reactions. Microorganisms obtain energy by transferring electrons from an electron donor such as an organic compound (or sometimes a reduced inorganic compound such as sulfide) to an electron acceptor. Electron acceptors are compounds that are relatively oxidized and include oxygen, nitrate, iron(III), manganese(IV), sulfate, carbon dioxide, or in some cases chlorinated aliphatic hydrocarbons such as tetrachloroethene (PCE), TCE, DCE and vinyl chloride (VC).

*Electron donor:* A compound capable of supplying (giving up) electrons during oxidation-reduction reactions. Microorganisms obtain energy by transferring electrons from an electron donor such as an organic compound (or sometimes a reduced inorganic compound such as sulfide) to an

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electron acceptor. Electron donors are compounds that are relatively reduced and include fuel hydrocarbons and native organic carbon.

*Metabolic byproduct:* A product of the reaction between an electron donor and an electron acceptor. Metabolic byproducts include volatile fatty acids, daughter products of chlorinated aliphatic hydrocarbons, methane, and chloride.

*Oxic groundwater:* Groundwater that contains oxygen in concentrations greater than about 0.5 mg/L.

*Oxidation/reduction reaction:* A chemical or biological reaction wherein an electron is transferred from an electron donor (donor is oxidized) to an electron acceptor (acceptor is reduced).

*Predominant terminal electron-accepting process:* The electron-accepting process (oxygen reduction, nitrate reduction, iron(III) reduction, etc.) that sequesters the majority of the electron flow in a given system.

*Reductive dechlorination:* Reduction of a chlorine-containing organic compound via the replacement of chlorine with hydrogen.

*Respiration:* The process of coupling the oxidation of organic compounds with the reduction of inorganic compounds such as oxygen, nitrate, iron(III), manganese(IV), and sulfate.

*Seepage velocity:* The average velocity of groundwater in a porous medium.

*Substrate:* A compound used by microorganisms to obtain energy for growth. The term can refer to either an electron acceptor or an electron donor.

#### 4.0 RESPONSIBILITIES

Project Manager (PM) / Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this standard operating procedure (SOP).

Project Hydrogeologist or Geochemist - Responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), and equipment to be used, and providing detailed input in this regard to the project plan documents. The project hydrogeologist or geochemist is also responsible for properly briefing and overseeing the performance of the site sampling personnel.

Site Manager (SM) / Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Project Geologist - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field sampling technicians or site personnel).

#### 5.0 PROCEDURES

##### 5.1 General

Natural attenuation includes physical, chemical, and biochemical processes affecting the concentrations of dissolved contaminants in groundwater. These processes may include advection, dispersion, volatilization, dilution, sorption to aquifer solids, and/or precipitation or mineralization of compounds. Of greatest importance are those processes that lead to a reduction in contaminant mass (by degrading or destroying contaminants) such as biodegradation. These biochemical processes remove organic contaminants from the aquifer by destruction. Depending on the type of contaminant, particularly the organic contaminant (e.g., petroleum hydrocarbons or chlorinated organic solvents), the biochemical environment in the aquifer will vary. The biochemical environment within the aquifer influences and is influenced by the activities of aquifer microbiota. Specific types of microbiota, working singly or in complex consortia, may use organic contaminants as part of their normal cell functions. Natural

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attenuation monitoring is designed to measure indicators of the biochemical environment within the aquifer and, with direct and indirect lines of evidence and associated chemical concentration data, evaluate the likely fate (i.e., transformation, destruction, dilution, attenuation, etc.) of organic contaminants.

## **5.2 Planning for Natural Attenuation Sampling**

The first step in preparing a natural attenuation investigation is to develop a site-specific conceptual model. The first step in development of this model is the analysis and review of available site-specific characterization data. The development and refinement of this model should be supplemented with additional data as needed. The data should include but is not limited to:

- Geologic and hydrogeologic information in three dimensions
- Nature, extent, and magnitude of contamination
- Location and presence of potential receptors to contamination

### **Lines of Evidence**

Several lines of evidence are used to determine whether natural attenuation is working. The most compelling, primary evidence is decreasing groundwater contaminant concentrations over time. Decreasing concentration trends can be demonstrated in several ways including:

- Isoconcentration maps of the dissolved plume over time wherein the extent of the plume is either stable or decreasing.
- Time series plots of contaminant concentrations within a well illustrating a clear downward trend.
- Contaminant concentration profiles in a series of monitoring wells along a groundwater flow path illustrating decreasing concentrations beyond that attributable to dilution and dispersion.

Secondary, or supporting, lines of evidence include:

- Analytical data showing production and subsequent destruction of primary contaminant breakdown products.
- Geochemical data indicating that the biochemical environment is favorable for the appropriate microbiota.
- Geochemical data that indicate the aquifer microbiota are active.

### **Monitoring Well Location and Sampling Frequency**

The number and locations of wells required to monitor natural attenuation will depend on the physical setting at each location. One possible array of monitoring wells is illustrated in Attachment A. In this scenario, one well is used to monitor conditions upgradient of the source, one well is located in the source area, and several wells are used to define and monitor the downgradient and lateral extent of the dissolved plume. At a minimum, there should be at least one upgradient well (ideally with no contamination present), one well in the source area, one well downgradient from the source area in the dissolved plume, and one downgradient well where contaminant concentrations are below regulatory criteria. Note that the number and locations of monitoring wells will vary depending on the site complexity and site objectives.

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Sampling frequency will be dictated by the ultimate use of the data and site-specific characteristics. Contaminant concentrations may be used to define statistically meaningful trends in contaminant concentrations. The sampling frequency may be defined by the hydrogeologic and/or geochemical conditions as well as the proposed statistical method for data analysis. For example, groundwater flow and contaminant characteristics (e.g., seepage velocity and contaminant loading) may dictate the sample frequency. Regardless of the factors, sampling frequency and duration will need to establish the range of natural chemical variability within the aquifer. After a sufficient amount of data has been collected and the geochemical conditions are understood, the frequency of sampling may be reduced. See Section 5.4 for additional information on sample collection and frequency.

### 5.3 Selection of Natural Attenuation Parameters

Natural attenuation via biodegradation depends on the nature of the organic contaminants and the oxidation-reduction (redox) environment within the aquifer. Simply stated, if the contaminants are fuels, biodegradation will be most effective if the redox conditions are aerobic or oxidizing. If the contaminants are chlorinated solvents, the biodegradation will be most effective (in the source and near source areas) if redox conditions in the aquifer are anaerobic or reducing.

Several parameters are needed to evaluate whether natural attenuation is taking place and, if so, the rate at which it may be occurring. The primary parameter providing direct evidence of natural attenuation is the aqueous concentrations of parent and daughter volatile organic compounds. More specifically, a decrease in parent products, an increase in daughter products, evidence that the plume is stable or shrinking in size, and overall decline in contaminant concentrations is direct evidence of natural attenuation. Natural attenuation or geochemical parameters that provide information about the redox conditions in the aquifer include:

- Dissolved oxygen
- Nitrate/nitrite
- Dissolved manganese
- Iron
- Sulfate/sulfide
- Methane
- Oxidation-reduction potential (ORP)

Secondary parameters that indicate biological activity in the aquifer and thereby support the natural attenuation evaluation include:

- Dissolved hydrogen
- Alkalinity
- Dissolved carbon dioxide

The concentrations of natural attenuation parameters are used to define the aquifer redox conditions. It is important to record and document the presence or absence (i.e., measurable or not measurable concentration) of certain natural attenuation parameters. The presence or absence of a certain substance may be sufficient to indicate the redox condition within the aquifer. By reference to Attachment B, which illustrates the typical sequence of biologically mediated redox reactions in natural systems, it is apparent that, for example, sulfate reduction (producing dissolved sulfide in groundwater) does not operate in an aerobic environment. Therefore, measurable sulfide should not be present if there is also dissolved oxygen at concentrations indicating an aerobic environment. Attachment B also illustrates the redox potential (measured in millivolts) associated with the redox reactions. ORP readings, also in millivolts, measured during well purging, may be compared with the range of values in Attachment B but with caution. Redox potentials measured with a platinum electrode in natural water samples may be misleading, especially when biologically mediated reactions are important, because many of the critical

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reactions in Attachment B do not generate a response in the electrode. Dissolved hydrogen concentration ranges associated with important redox reactions are also indicated in Attachment B. Because dissolved hydrogen is actually used by microbiota during redox reactions, its concentration may provide an additional indicator of the overall redox condition in the aquifer.

Attachments C and D tabulate the natural attenuation parameters for chlorinated volatile organic compound and petroleum hydrocarbon plumes, respectively. The parameters listed in these tables are organized in order of importance. Parameters selected for analysis shall be determined based on site conditions, project-specific plans, and/or other criteria established for the project. Based on these criteria, it is possible that all of the parameters may be selected.

#### **5.4 Selection of Natural Attenuation Analytical Methods and Procedures**

There are many analytical methods available to measure concentrations of the natural attenuation parameters discussed in the previous sections. Attachment E summarizes the sample methodologies, sampling equipment needed, sample volume, container, preservation, and holding time requirements. This table also summarizes the detection limits and the detection ranges for each method. A number of factors should be considered when selecting the appropriate sample analytical methodology including the required parameters, appropriate detection ranges for each compound, cost, and ease of use in the field. For example, when determining the correct methodology for measuring concentrations of total sulfide, the metabolic byproduct of sulfate reducing conditions, it is important to analyze for each of the forms of sulfide ( $H_2S$ ,  $S^{2-}$ , and  $HS^-$ ). Also, when the detection limit of the selected method is exceeded, another method may be considered, or the sampler may be able to dilute the sample (per manufacturer's instructions) to quantify it within the detected range. In terms of cost, some parameters are very time consuming when performed in the field. Without sacrificing sample integrity it may be more appropriate to select a methodology performed in a fixed-base laboratory. Finally, in terms of ease of use, certain field methods are generally easier compared to other methods. Using simpler methods may result in better quality sample results and increased sample repeatability without sacrificing sample integrity. For example, in some cases CHEMetrics Titret® Titration Ampule kits may be a good alternative to other hand digital titration methods.

The sample technicians should be aware that based on geochemical conditions recorded in the field, certain geochemical parameters may not have positive detections. For example, if dissolved oxygen concentrations indicate aerobic conditions then it is unlikely that dissolved hydrogen is present (see Section 5.10 for additional information). Another example is alkalinity. If the pH of the groundwater sample is less than 4.5, then it is unlikely that alkalinity will be measurable. Despite the potential for non-detect results, in cases such as those described above, all parameters should be collected in the field based upon project plans. The value in collecting the parameters in the future shall be determined by the project hydrogeologist and/or geochemist in accordance with the projects planning documents data quality objectives (DQO) and the items discussed in Section 5.2.

#### **5.5 Procedures for Sample Collection**

Groundwater sample collection for natural attenuation sampling should be performed using low flow purging and sampling techniques. These techniques are described in detail in SOP SA-1.1. Low flow purging and sampling procedures should be used to ensure the collection of a sample that is "representative" of the water present in the aquifer formation. Minimizing stress on the aquifer formation during low flow purging and sample collection ensures that there are minimal alternations to the water chemistry of the sample. The criteria used in the purging process should include minimization of drawdown in the well, stabilization of applicable indicator parameters, and evacuation of a sufficient amount of purge volume in accordance with SOP SA-1.1, project plans, and/or applicable regulatory guidance.

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Groundwater purging and sampling for natural attenuation should be performed using submersible pumps (e.g., bladder pumps) in accordance with SOP SA-1.1. However, in accordance with project plans and applicable regulatory guidance, peristaltic pumps may also be used for this purpose. Limitations of and factors associated with using these devices should be considered (see SOP SA-1.1 for more information). As a result of difficulties in collecting "representative" groundwater samples, bailers should not be used for the collection of natural attenuation samples.

It is critical that disturbance and aeration of samples monitored and collected at the well head are minimized. As a result, a flow-through sampling cell and a direct reading meter shall be used for the measurement of well stabilization indicator parameters (e.g., pH, conductivity, temperature, dissolved oxygen, turbidity, and ORP) at the well head. The pump effluent tubing should be placed at the bottom of the flow-through cell allowing effluent water from the cell to discharge at the top of the meter (above the detector probes) to minimize the agitation of water in the cell.

Documentation of the purging process shall be recorded during and at the completion of purging as discussed in Section 5.8. Immediately following the purging process and before sampling, all applicable indicator parameters must be measured and recorded on the appropriate sample log sheets as discussed in Section 5.8.

After all of the purging requirements have been met, groundwater sampling and natural attenuation data collection can begin. Monitoring wells will be sampled using the same pump and tubing used during well purging.

## **5.6 Procedures for Field Sample Analysis**

Each of the field and fixed-base laboratory sample parameters requires different sampling procedures and holding times. Attachment E presents parameter-specific requirements for sampling, analysis, and storage of all of the parameters and methods sampled as part of natural attenuation analysis.

Due to parameter procedure and holding times, it is important to consider the sequence of sample collection and analysis. Generally speaking, with the exception of volatile organic compounds, field parameters shall be analyzed first followed by fixed-base laboratory sample collection. All samples will be collected in a sequence and manner that minimizes volatilization, oxidation, and/or chemical transformation of compounds. As a result, the following sample and analysis order should be followed:

- |   |                                    |
|---|------------------------------------|
| 1. Volatile organic compounds                       | 8. Nitrate / Nitrite               |
| 2. Dissolved oxygen                                 | 9. Dissolved manganese             |
| 3. Alkalinity                                       | 10. Semivolatile organic compounds |
| 4. Dissolved carbon dioxide                         | 11. Other dissolved metals         |
| 5. Dissolved ferrous iron                           | 12. Total metals                   |
| 6. Dissolved sulfide (hydrogen sulfide and sulfide) | 13. All other constituents         |
| 7. Dissolved hydrogen, methane, ethene, and ethane  |                                    |

Field-analyzed parameters should be collected and immediately analyzed directly from the pump effluent per the requirements on Attachment E and manufacturer's recommendations. Care should be taken to minimize any unnecessary disturbance, aeration, or agitation of the sample prior to analysis. It is not acceptable to collect and store samples that are to be analyzed immediately at the well head in a temporary holding container (e.g., open topped pitcher) to be analyzed at a later time.

The manufacturer's procedure manual for each of the field-based analyses shall be maintained in the field during the entire sampling program. The procedures give a detailed explanation of how to perform each particular method and include information on sampling, storage, accuracy checks, interferences, reagents, and apparatus needed to perform each analysis.

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### 5.7 Procedures for Quality Assurance and Quality Control Field Sample Analysis

Accuracy and precision checks shall be performed to check the performance of the reagents, apparatus, and field analytical procedures per the manufacturer's recommendations. The accuracy checks should include the use of standard solutions (i.e., standard addition), as appropriate. The manufacturer's field test kit manual provides details on how to perform each of the accuracy checks for each parameter where applicable. Refer to Section 6.0 for manufacturer contact information.

Precision checks must include the performance of duplicate analysis. When using a colorimeter, precision checks may also include reagent blank corrections and standard curve adjustments as recommended by the manufacturer. Field duplicate results shall be performed and evaluated for relative percent difference (RPD) at a rate of 1 per 10 samples or as determined by the project plans. The RPD can be calculated as follows:

$$RPD = \left| \frac{\text{First result} - \text{Second result}}{\text{Mean arithmetic (average) of first and second result}} \right| \times 100$$

If the RPD exceeds 50 percent, it is required that the test be performed again to verify the result. The duplicate results shall be documented in the 'Notes' section for that specific parameter on the appropriate sample logsheet (see Section 5.8).

If a colorimeter (e.g., HACH DR-890 or equivalent) is used for parameter analysis, an instrument performance verification test using absorbance standards may also be performed to ensure the meter is providing accurate measurements.

The following table lists examples of the types and frequencies of accuracy checks required for each parameter. Refer to the manufacturer's instructions for information regarding other analyses.

Parameter	Method	Standard Solution	Field Duplicate	Reagent Blank Correction
Alkalinity	CHEMetrics K-9810, -15, -20	None	1 per 10	None
Carbon dioxide	CHEMetrics K-1910, -20, -25	None	1 per 10	None
Dissolved oxygen	CHEMetrics K-7501, -12	None	1 per 10	None
Ferrous iron	HACH DR-890	None	1 per 10	None
Nitrite	HACH DR-890	1 per round	1 per 10	1 per lot
Nitrate	HACH DR-890	1 per round	1 per 10	1 per lot
Sulfide	HACH DR-890	None	1 per 10	None
Hydrogen sulfide	HACH HS-C	None	1 per 10	None

Prior to analysis, the expiration dates of reagents shall be checked. If the reagents have exceeded their expiration date or shelf life, the reagents shall be replaced. If deviations from the applicable analytical procedure are identified, the deviations shall be corrected and the associated samples re-analyzed. If problems are identified with the reagents, apparatus, or procedures, data interferences may be present. Interferences may also be due to other factors (e.g., pH, presence or concentration of other ions, turbidity, temperature, etc.) that may interfere with the sample result. The manufacturer's procedures (e.g., Hach, 1999) should be reviewed prior to analysis to avoid or minimize such interferences. Associated problems

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or suspected interferences shall be documented in the 'Notes' section of the sample logsheet. Often, interferences cannot be avoided. In these cases, the sampler should be aware of these potential interferences and document them properly.

### **5.8 Documentation Procedures for Field Sample Analysis**

Field results shall be properly documented in the field as noted in SOP SA-6.3. The sample log sheet titled "Field Analytical Log Sheet, Geochemical Parameters" shall be prepared for each sample collected and analyzed in the field. A copy of this form can be found as Attachment F of this SOP. Other field log sheets (e.g., low flow purge log sheet, groundwater sample logsheet, etc.) shall also be completed in accordance with SOP SA-6.3.

Specific information shall also be recorded in the project logbook. This information shall include, but is not limited to, the test kit name and model number, lot number and expiration date of the test kit and reagents used, serial number of the instrument (e.g., colorimeter) used for the analysis, and results of the quality assurance and quality control field sample analysis. Because environmental conditions and changes in those conditions may affect the field analytical results, it is important to document the site conditions (weather, temperature, etc.) at the time of sampling in the logbook in accordance with SOP SA-6.3.

### **5.9 Waste Handling and Disposal**

Several of the test kits listed in Attachment E require the use of chemicals and materials that must be properly handled and disposed of in a proper and responsible manner. Refer to specific manufacturer's guidance for handling and disposal practices. See also Section 6.0 for more detailed and complete information. Handling and disposal of these items should be conducted in accordance with all local, state, and federal guidelines.

### **5.10 Understanding Field Sample Analytical Results**

Natural attenuation data interpretation is complicated by the complex inter-relationships of various parameters. The complexity reflects the myriad of biochemical processes. Real-time evaluation of field analytical data can be misleading because a full interpretation often requires combining the field analytical results with fixed-base laboratory results. Regardless, some simple observations and data interpretations in the field may provide insights about the monitoring system or early warnings about sample collection and handling problems.

Data collected from the designated upgradient monitoring well is the baseline from which other interpretations are made. Field analytical data will indicate that the upgradient environment is either oxidizing or reducing. The redox condition within the upgradient area of the aquifer may be natural or impacted by other contaminant source areas (see Section 5.2 for upgradient well selection). Regardless, the redox condition of the upgradient groundwater will influence the source area. Changes in field analytical results from the upgradient well to the source area well will be reflected in samples from monitoring wells further downgradient.

The general characteristics of the two redox environments are summarized in the following table.

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Aerobic/Oxidizing	Anaerobic/Reducing
<ul style="list-style-type: none"> <li>• Measurable dissolved oxygen (&gt;1 to 2 ppm)</li> <li>• Measurable nitrate</li> <li>• No measurable dissolved manganese</li> <li>• No measurable dissolved ferrous iron</li> <li>• Measurable dissolved sulfate</li> <li>• No measurable dissolved sulfide</li> <li>• No measurable dissolved methane</li> <li>• No measurable dissolved hydrogen</li> </ul>	<ul style="list-style-type: none"> <li>• No measurable dissolved oxygen (&lt;1 ppm)</li> <li>• No measurable nitrate</li> <li>• Measurable dissolved manganese</li> <li>• Measurable dissolved ferrous iron</li> <li>• No measurable dissolved sulfate</li> <li>• Measurable dissolved sulfide</li> <li>• Measurable dissolved methane</li> <li>• Measurable dissolved hydrogen</li> </ul>

Transitional environments between these two extremes may have intermediate characteristics and are actually quite common. Because reactions are mediated by biological systems, equilibrium (the basis for the figure in Attachment B) conditions within the aquifer should not be expected. For example, sulfate reduction environments may occur in close proximity to methanogenic environments, and this natural attenuation data may be difficult to interpret. Carefully collected and analyzed field measurements and sample collections for fixed-base laboratory analyses are designed to characterize the aquifer environment along the continuum between strongly aerobic and strongly anaerobic. Because the land surface environment is generally more oxidizing than any groundwater environment, sample handling at the point of collection and analysis is extremely important in preserving the chemical integrity of the groundwater sample.

## 6.0 REFERENCES

American Society for Testing and Materials (ASTM), 1998. Standard Guide for Remediation of Ground Water by Natural Attenuation at Petroleum Release Sites, Designation: E1943-98, West Conshohocken, Pennsylvania.

Chemetrics, 2002, <http://www.chemetrics.com>.

Department of the Navy, 1998. Technical Guidelines for Evaluating Monitored Natural Attenuation of Petroleum Hydrocarbons and Chlorinated Solvents in Ground Water at Naval and Marine Corps Facilities, Department of the Navy, September. Prepared by T. H. Weidemeier and F. H. Chappelle.

USEPA (United States Environmental Protection Agency), 1998. Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Ground Water, EPA/600/R-98/128, Office of Research and Development, Washington, D.C.

Hach Company, 1999. DR-890 Colorimeter Procedures Manual, Product Number 48470-22, Loveland Colorado.

Hach Company, 1999. Digital Titrator (manual), Model Number 16900, Catalog Number 16900-08. Loveland, Colorado.

Hach Company, 2002, <http://www.hach.com/>.

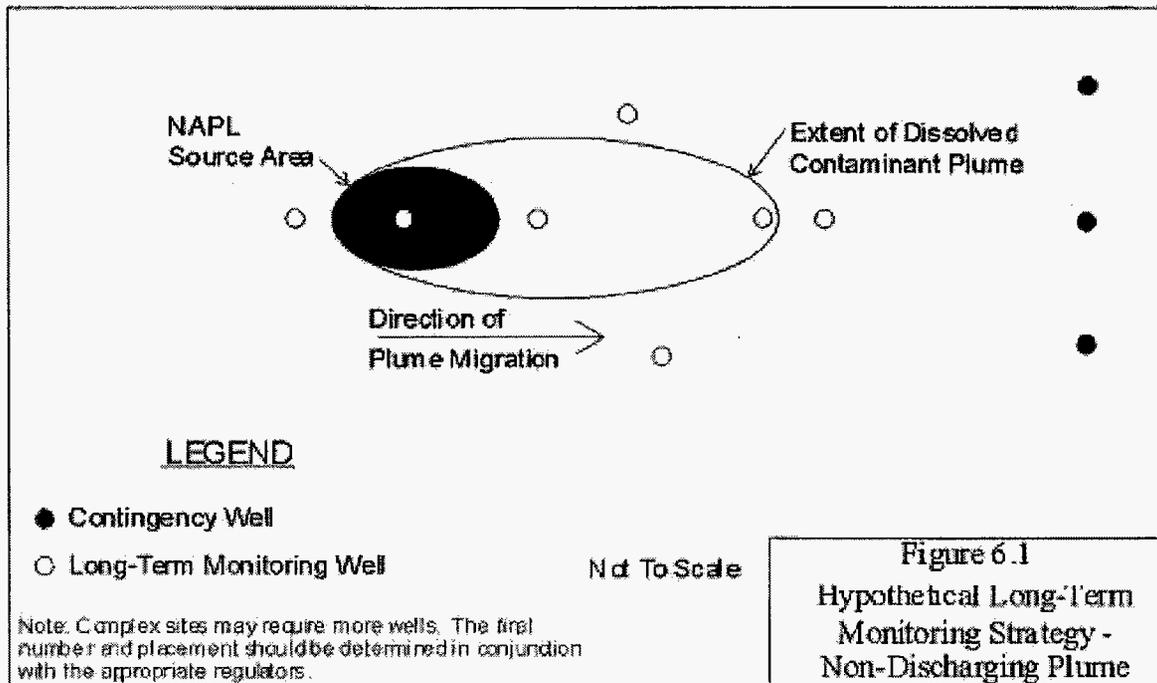
USEPA, 1997. Draft EPA Region 4 Suggested Practices for Evaluation of a Site for Natural Attenuation (Biological Degradation) of Chlorinated Solvents; Version 3.0. November.

USEPA, 1999. Use of Monitored Natural Attenuation at Superfund, RCRA Corrective Action, and Underground Storage Tank Sites, USEPA OSWER Directive 9200.4-17P, April 21, 1999

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**ATTACHMENT A**

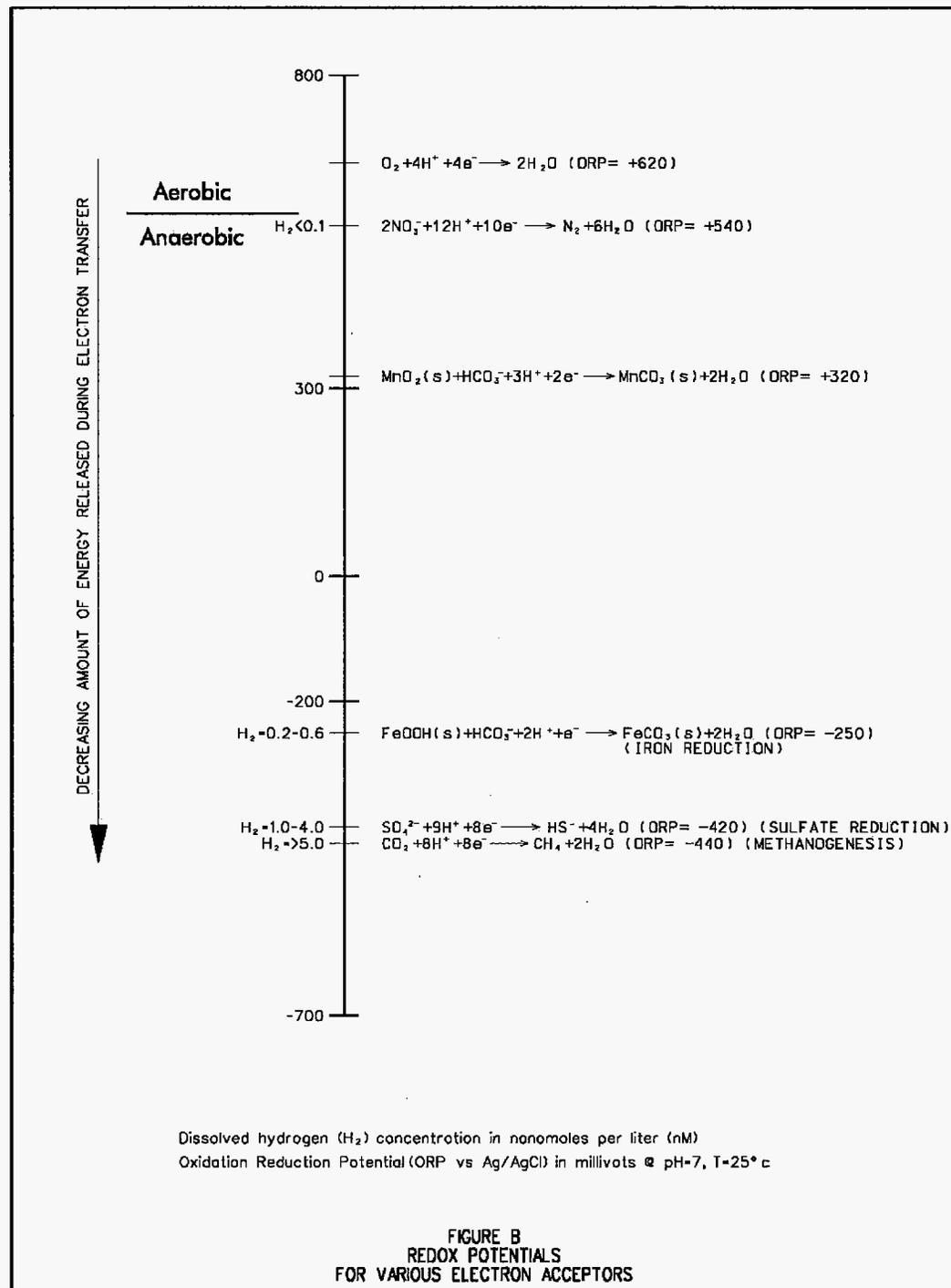
**HYPOTHETICAL LONG-TERM MONITORING STRATEGY**



Taken from: Department of the Navy, 1998, Technical Guidelines for Evaluating Monitored Natural Attenuation of Petroleum Hydrocarbons and Chlorinated Solvents in Ground Water at Naval and Marine Corps Facilities, Prepared by Todd Weidemeier and Francis Chappelle.

## ATTACHMENT B

## REDOX POTENTIALS FOR VARIOUS ELECTRON ACCEPTORS



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### ATTACHMENT C

#### NATURAL ATTENUATION PARAMETERS FOR CHLORINATED VOLATILE ORGANIC COMPOUND PLUMES SCREENING PROCESS SUMMARY FOR REDUCTIVE (ANAEROBIC) DECHLORINATION

Potential Electron Donors	Electron Acceptors:	Reduced Species:	Related Dechlorination Pathway:
Native total organic carbon (TOC) Anthropogenic carbon (e.g., leachate) Fuel hydrocarbons (e.g., BTEX) Lightly chlorinated solvents (DCE/VC)	Dissolved Oxygen	⇒ Carbon Dioxide (CO <sub>2</sub> )	~ DCE → VC → CO <sub>2</sub>
	Manganese (Mn <sup>4+</sup> )	⇒ Manganese (Mn <sup>2+</sup> )	~ DCE → VC
	Nitrate (NO <sub>3</sub> )	⇒ Nitrite (NO <sub>2</sub> )	~ DCE → VC
	Ferric Iron (Fe <sup>3+</sup> )	⇒ Ferrous Iron (Fe <sup>2+</sup> )	~ DCE → VC → CO <sub>2</sub>
	Sulfate (SO <sub>4</sub> )	⇒ Sulfide (S <sup>2-</sup> , HS <sup>-</sup> , H <sub>2</sub> S)	~ TCE → DCE → VC → Ethene
	Carbon Dioxide (CO <sub>2</sub> )	⇒ Methane (CH <sub>4</sub> )	~ PCE → TCE → DCE → VC → Ethene

#### Geochemical Parameter List:

Parameter	Field or Lab	Rationale	Importance
Volatile organic compounds	L	Source products; daughter products; electron donors (e.g., benzene, toluene, ethylbenzene, and xylene; BTEX)	1
Dissolved oxygen	F	Primary electron acceptor (respiration); an/aerobic indicator	1
Nitrate (and nitrite), dissolved	F or L	Anaerobic electron acceptor (product of nitrate reduction)	1
Manganese, dissolved	F or L	Anaerobic electron acceptor	1
Ferrous Iron (Fe <sup>2+</sup> )	F	Product of iron reduction	1
Sulfate [and sulfide (S <sup>2-</sup> )]	F or L	Common anaerobic electron acceptor (product of sulfate reduction)	1
Sulfide (H <sub>2</sub> S)	F	Common product of sulfate reduction	1
Methane, ethane, ethene	L	Product of methanogenesis; daughter products of reductive dechlorination	1
Chloride	L	Ultimate daughter product of reductive dechlorination	1
TOC - upgradient groundwater	L	Electron donor	1
ORP, pH, specific conductance, temperature, turbidity	F	General water quality determination	1
Carbon dioxide (CO <sub>2</sub> )	F	Anaerobic electron acceptor (methanogenesis); biotic respiration indicator	2
Alkalinity/DIC	F	Buffering capacity; biotic respiration indicator	2
Hydrogen, dissolved	L	Fingerprint for characterizing electron acceptor pathway - indicator of what redox is occurring	2
TOC - upgradient soil	L	Input to analytical NA models; quantifies soil-water distribution coefficient and retardation factor	2
Volatile fatty acids	L	Determination of anthropogenic carbon used as an electron donor	3

Importance: 1=Most important; 3=Least important (depending on DQOs, all may be recommended). See Attachment E for details regarding analytical methods.

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**ATTACHMENT D**

**NATURAL ATTENUATION PARAMETERS FOR  
PETROLEUM HYDROCARBON PLUMES  
SCREENING PROCESS SUMMARY FOR OXIDATIVE (AEROBIC) DEGRADATION**

Parameter	Field or Lab	Rationale	Importance
Volatile organic compounds	L	Source products; daughter products; electron donors (BTEX)	1
Dissolved oxygen	F	Primary electron acceptor (respiration); an/aerobic indicator	1
Nitrate (and nitrite), dissolved	F or L	Anaerobic electron acceptor (and product of nitrate reduction)	1
Manganese, dissolved	F or L	Anaerobic electron acceptor	1
Ferrous Iron (Fe <sup>2+</sup> )	F	Product of iron reduction	1
Sulfate [and Sulfide (S <sup>-2</sup> )]	F or L	Common anaerobic electron acceptor (product of sulfate reduction)	1
Sulfide (H <sub>2</sub> S)	F	Common product of sulfate reduction	1
TOC - upgradient groundwater	L	Electron donor	1
ORP, pH, specific conductance temperature, turbidity	F	General water quality determination	1
Dissolved methane (CH <sub>4</sub> )	L	Product of methanogenesis	1
Anions: chloride (Cl), nitrate (NO <sub>3</sub> ), nitrite (NO <sub>2</sub> ), phosphate (PO <sub>4</sub> ), sulfate (SO <sub>4</sub> )	L		1
TOC - Upgradient soil	L	Input to analytical NA models; quantifies soil-water distribution coefficient and retardation factor	2
Biological oxygen demand (BOD)	L	Understanding of aquifer oxygen demand	3
Chemical oxygen demand (COD)	L	Understanding of aquifer oxygen demand	3

Importance: 1=Most important; 3=Least important (depending on DQOs, all may be recommended).

See Attachment E for details regarding analytical methods.

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**ATTACHMENT E**  
**GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUMES, CONTAINERS,**  
**PRESERVATION, HOLDING TIMES, AND DETECTION RANGES**  
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Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Alkalinity	CHEMetrics K-9810, K-9815, K-9820 -ASTM D 1067-92 -EPA 310.1	Titret® Titration Ampules / Hydrochloric Acid, Phenolphthalein	Field. Follow test kit instructions. Avoid agitation and analyze at well head to determine total alkalinity. Filter if turbid (>10 NTU).	10-100 (K-9810) 50-500 (K-9815) 100-1000 (K-9820)	N/A	10 50 100-
Alkalinity	Fixed-base lab -EPA 310.1	N/A	100 to 250 mL in glass or plastic container. Cool to 4°C. Analyze within 14 days. Filter if turbid.	N/A	N/A	N/A
Alkalinity / Dissolved Inorganic Carbon	HACH AL-DT -HACH 8203 -SM 2320 / SM 403	Digital Titration / Hydrochloric Acid, Phenolphthalein (P) and Total (M)	Field. Follow test kit instructions. Avoid agitation and analyze at well head to determine carbonate, bicarbonate, and hydroxide ions. Filter if turbid as recommended by manufacture. May use a pH meter for colored samples.	10-4000	N/A	10
Arsenic	Fixed-base lab -SW-6010 B	N/A	1 liter glass or polyethylene container, HNO <sub>3</sub> to pH ≤ 2. 6 months.	N/A	N/A	N/A
Biochemical Oxygen Demand	Fixed-base lab -EPA 410.1	N/A	2 liter HDPE. Cool to 4°C. Analyze within 48 hours.	N/A	N/A	N/A
Carbon Dioxide, dissolved	CHEMetrics K-1910, K-1920, K-1925 -ASTM D 513.82 -SM 4500-CO <sub>2</sub> -C	Titret® Titration Ampules / Sodium Hydroxide, Phenolphthalein	Field. Follow test kit instructions. Avoid agitation and analyze at well head.	10-100 (K-1910) 100-1000 (K-1920) 250-2500 (K-1925)	N/A	10 100 250
Carbon Dioxide, dissolved	Fixed-base lab -VOA water sample (Vaportech)	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Carbon Dioxide, dissolved	Fixed-base lab -Microseeps gas stripping cell	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Carbon Dioxide, dissolved	HACH CA-DT -HACH 8205 -Mod. SM 406	Digital Titration / Sodium Hydroxide, Phenolphthalein	Field. Follow test kit instructions. Do not aerate or agitate. Analyze at well head.	10-1000	N/A	10
Chemical Oxygen Demand	Fixed-base lab -EPA 410.1	N/A	125 mL HDPE. H <sub>2</sub> SO <sub>4</sub> to pH <2.0. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Chloride (Cl)	Fixed-base lab -EPA 300	N/A	100 to 250 mL in glass or plastic container. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Chlorine - Total (Cl <sub>2</sub> )	HACH DR-850 -HACH 8167 -SM 4500-Cl	Colorimeter / DPD Method	Field. Follow test kit instructions.	0.02-2.00	± 0.01 mg/L with a 1.00 mg/L chlorine solution.	1
Conductance, Specific	Field Meter -SW-9050 A	Direct Reading Meter	100 to 250 mL in glass or plastic container. Analyze immediately.	N/A	N/A	N/A
Ethane, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Ethane, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A

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## ATTACHMENT E

GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,  
PRESERVATION, HOLDING TIME, AND DETECTION RANGES  
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Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Ethene, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Ethene, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Fraction Organic Carbon (foc)-Soil Upgradient Saturated Soil	Fixed-base lab -Walk-Black -SW-846 9060	N/A	200 gram glass jar. Cool to 4°C. Analyze within 14 days.	N/A	N/A	N/A
Hydrogen, dissolved	Fixed-base lab -Microseeps or Vapor Tech gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial.	N/A	N/A	N/A
Iron, ferrous (Fe <sup>2+</sup> )	HACH DR-850 -HACH 8146 -Mod. SM 315 B	Colorimeter 1, 10 Phenanthroline	Field. Follow test kit instructions. Analyze immediately at well head. Filter if turbid (>10 NTU) as recommended by the manufacture.	0-3.00	±0.017 mg/L with a 2.00 mg/L Fe <sup>2+</sup> solution.	0.03
Iron, ferrous (Fe <sup>2+</sup> )	HACH IR-18C -Mod. SM 315 B	Color Disc 1, 10 Phenanthroline	Field. Follow test kit instructions. Analyze immediately at well head. Filter if turbid (>10 NTU) as recommended by the manufacture.	0-10	N/A	0.2
Iron, total dissolved (Filtered)	Fixed-base lab -SW-846 6010B	N/A	250 mL in plastic container. Field filter to 0.45 µ. HCl to pH <2. Cool to 4°C. Analyze within 6 months.	N/A	N/A	N/A
Manganese (Mn <sup>2+</sup> )	HACH DR-850 -HACH 8034 -CFR 44(116) 34193	Colorimeter / Cold Periodate Oxidation	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-20.0	+ 0.19 mg/L with a 10.00 mg/L Mn solution.	0.12
Manganese (Mn <sup>2+</sup> )	HACH MN-5 -Mod. SM 319 B -CFR 44(116) 34193	Color Disc / Cold Periodate Oxidation	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-3	N/A	0.1
Manganese, total dissolved (Filtered)	Fixed-base lab -SW-846 6010B	N/A	250 mL in plastic container. Field filter to 0.45 µ. HCl to pH <2. Cool to 4°C. Analyze within 6 months.	N/A	N/A	N/A
Methane, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Methane, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Nitrate (NO <sub>3</sub> )	Fixed-base lab -EPA 300	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours.	N/A	N/A	N/A
Nitrate (NO <sub>3</sub> )	HACH DR-850 -HACH 8192 -Mod. EPA 353.2	Colorimeter / Cadmium Reduction	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Pretreatment required if nitrite is present.	0-0.50	± 0.03 mg/L with a 0.25 mg/L of nitrate nitrogen (NO <sub>3</sub> <sup>-</sup> N) solution.	0.01
Nitrite (NO <sub>2</sub> )	Fixed-base lab -EPA 300	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A

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**ATTACHMENT E**

**GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,  
 PRESERVATION, HOLDING TIME, AND DETECTION RANGES  
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Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Nitrite (NO <sub>2</sub> <sup>-</sup> )	HACH DR-850 -HACH 8507 -Mod. EPA 354.1 -Mod. SM 419 -CFR 44(85) 25595	Colorimeter / Diazotization	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-0.350	± 0.001 mg/L with a 0.250 mg/L nitrite nitrogen solution.	0.005
Nitrogen, dissolved	Fixed-base lab -Microseeps gas stripping cell -Vaportech VOA water sample	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required for Microseeps. Ship in glass septum vial (Microseeps) or VOA vial (Vaportech).	N/A	N/A	N/A
Nitrogen, Total Kjeldahl	Fixed-base lab -EPA 351.2	N/A	500 mL plastic/glass container. Cool to 4°C. H <sub>2</sub> SO <sub>4</sub> to pH ≤ 2. Analyze within 28 days.	N/A	N/A	N/A
Oxidation Reduction Potential	Field Meter - ASTM D-1498	Direct Reading Meter	Field. Do not aerate. Gently agitate probe using flow over or flow-through method. Analyze immediately at well head.	N/A	N/A	N/A
Oxygen, dissolved	CHEMetrics K-7501, K-7512 -ASTM D 5543-94 -ASTM D 887-92	CHEMets® Vacuum Vials / Rhodazine D and Indigo Carmine	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-1 (K-7501) 1-12 (K-7512)	N/A	0.025 1
Oxygen, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Oxygen, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Oxygen, dissolved	HACH OX-DT HACH 8215 -SM 4500-O-G	Digital Titration / Azide Modification of Winkler Digital Titration Method	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	1-10	N/A	1
Oxygen, dissolved	HACH DR-850 (AccuVac Ampules) LR HRDO Method	-Indigo Carmine Method -Rhodazine D Method	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-0.8 ppm 0-10 ppm	0.01 ppm 0.1 ppm	N/A
Oxygen, dissolved	Field Meter	Direct Reading Meter	Analyze immediately at well head. Avoid agitation and analyze immediately at well head. Used for well stabilization measurement parameter only.	N/A	N/A	N/A
pH	Field Meter -SW 9040B	Direct Reading Meter	Analyze immediately at well head.	N/A	N/A	N/A
Phosphate (ortho)	Fixed-base lab -EPA 300	Ion Chromatography	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Phosphate, potassium	Fixed-base lab -SV-846 6010B	Inductively Coupled Plasma	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Salinity	Field Meter	Direct Reading Meter	Analyze immediately.	N/A	N/A	N/A
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	Fixed-base lab	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	HACH DR-850 -HACH 8051 -EPA 375.4	Colorimeter / Turbimetric SulfaVer 4	Field. Follow test kit instructions. Filter if turbid as recommended by the manufacture.	0-70	± 0.5 mg/L with a 50 mg/L sulfate solution.	4.9
Sulfide (Hydrogen Sulfide, H <sub>2</sub> S)	HACH HS-C -HACH Proprietary -Mod. SM 426 C	Color Chart / Effervescence of H <sub>2</sub> S through sulfide reactive paper.	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-5	N/A	0.1
Sulfide (S <sup>2-</sup> )	CHEMetrics K-9510 -SM 4500-S <sup>2</sup>	CHEMets® Vacuum Vials / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-1 1-10	N/A	0.1 1

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**ATTACHMENT E**

**GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,  
 PRESERVATION, HOLDING TIME, AND DETECTION RANGES  
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Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Sulfide (S <sup>2-</sup> )	Fixed-base lab -EPA 376.1/376.2	N/A	1 liter in plastic container, no headspace. NaOH to pH >9. Cool to 4°C. Avoid agitation and analyze within 7 days.	N/A	N/A	N/A
Sulfide (S <sup>2-</sup> )	HACH DR-850 -HACH 8131 -SM 4500-S <sup>2</sup>	Colorimeter / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head. Pretreatment required for turbid samples as recommended by the manufacture.	0-0.70	± 0.02 mg/L with a 0.73 mg/L sulfide solution.	0.01
Sulfide (S <sup>2-</sup> )	HACH HS-WR -SM 4500-S <sup>2</sup>	Color Disc / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head. Pretreatment required for turbid samples as recommended by the manufacture.	0-11.25	N/A	0.1-2.5
Temperature	Field Meter / Thermometer - E 170.1	Direct Reading Meter / Thermometer	Analyze immediately.	N/A	N/A	N/A
Total Organic Carbon (TOC)-Groundwater	Fixed-base lab -E 415.1	N/A	125 mL HDPE. H <sub>2</sub> SO <sub>4</sub> to pH < 2.0. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Turbidity	Field Meter - E 180.1	Direct Reading Meter	Analyze immediately.	N/A	N/A	N/A

N/A = Not applicable.

**ATTACHMENT F**

**FIELD ANALYTICAL LOG SHEET, GEOCHEMICAL PARAMETERS  
PAGE 1 OF 3**

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.



**FIELD ANALYTICAL LOG SHEET  
GEOCHEMICAL PARAMETERS**

Tetra Tech NUS, Inc.

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Project Site Name: _____		Sample ID No.: _____	
Project No.: _____		Sample Location: _____	
Sampled By: _____		Duplicate: <input type="checkbox"/>	
Field Analyst: _____		Blank: <input type="checkbox"/>	
Field Form Checked as per QA/QC Checklist (initials): _____			
<b>SAMPLING DATA:</b>			
Date: _____	Color (Visual)	pH (S.U.)	S.C. (mS/cm)
Time: _____			Temp. (°C)
Method: _____			Turbidity (NTU)
			DO (mg/l)
			Salinity (%)
			ORP (Eh) (+/- mv)
<b>SAMPLE COLLECTION/ANALYSIS INFORMATION:</b>			
<b>ORP (Eh) (+/- mv)</b>		Electrode Make & Model: _____	
Reference Electrode (circle one): Silver-Silver Chloride / Calomel / Hydrogen			
<b>Dissolved Oxygen:</b>			
Equipment: Chemetrics Test Kit		Concentration: _____ ppm	
Range Used:	Range	Method	Concentration ppm
<input type="checkbox"/>	0 to 1 ppm	K-7510	
<input type="checkbox"/>	1 to 12 ppm	K-7512	
Equipment: HACH Digital Titrator OX-DT		Analysis Time: _____	
Range Used:	Range	Sample Vol.	Cartridge
<input type="checkbox"/>	1-5 mg/L	200 ml	0.200 N
<input type="checkbox"/>	2-10 mg/L	100 ml	0.200 N
			Multiplier
			0.01
			0.02
Notes:		Titration Count	
		Multiplier	
		Concentration	
		x 0.01 = mg/L	
		x 0.02 = mg/L	
<b>Carbon Dioxide:</b>			
Equipment: Chemetrics Test Kit		Concentration: _____ ppm	
Range Used:	Range	Method	Concentration ppm
<input type="checkbox"/>	10 to 100 ppm	K-1910	
<input type="checkbox"/>	100 to 1000 ppm	K-1920	
<input type="checkbox"/>	250 to 2500 ppm	K-1925	
Equipment: HACH Digital Titrator CA-DT		Analysis Time: _____	
Range Used:	Range	Sample Vol.	Cartridge
<input type="checkbox"/>	10-50 mg/L	200 ml	0.3636 N
<input type="checkbox"/>	20-100 mg/L	100 ml	0.3636 N
<input type="checkbox"/>	100-400 mg/L	200 ml	3.636 N
<input type="checkbox"/>	200-1000 mg/L	100 ml	3.636 N
			Multiplier
			0.1
			0.2
			1.0
			2.0
Standard Additions: <input type="checkbox"/>		Titrant Molarity: _____	
		Digits Required: 1st: _____ 2nd: _____ 3rd: _____	
Notes:			
<b>Hydrogen, dissolved</b>			
Equipment: Bubble strip sampling field method			
Start stripper at _____ (time)			
End stripper at _____ (time)			
Total stripper time _____			
Pump rate _____ milliliters/minute			



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**ATTACHMENT F**

**FIELD ANALYTICAL LOG SHEET, GEOCHEMICAL PARAMETERS  
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Note: Analyte, method, and/or equipment may be deleted from form if not being performed.

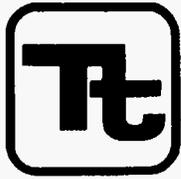


**FIELD ANALYTICAL LOG SHEET  
GEOCHEMICAL PARAMETERS**

Tetra Tech NUS, Inc.

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Project Site Name: _____		Sample ID No.: _____	
Project No.: _____		Sample Location: _____	
Sampled By: _____		Duplicate: <input type="checkbox"/>	
Field Analyst: _____		Blank: <input type="checkbox"/>	
<b>Sulfate (SO<sub>4</sub><sup>2-</sup>):</b>			
Equipment	DR-850	DR-8 __	Range: 0 - 70 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 91		
Analysis Time:	_____		
Standard Solution:	<input type="checkbox"/>	Results: _____	Filtered: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	
Notes: _____			
<b>Nitrate (NO<sub>3</sub><sup>-</sup>-N):</b>			
Equipment	DR-850	DR-8 __	Range: 0 - 0.50 mg/L <sup>(1)</sup>
Concentration:	_____ ppm		
Program/Module:	_____ 55		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Nitrite Interference Treatment: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	Reagent Blank Correction: <input type="checkbox"/>
Alternate forms: NO <sub>2</sub> _____ NaNO <sub>2</sub> _____ mg/L			
Notes (1): If results are over limit use dilution method at step 3, 5ml sample 10ml DI result X3, range upto 1.5mg/L			
Notes: _____			
<b>Nitrite (NO<sub>2</sub><sup>-</sup>-N):</b>			
Equipment	DR-850	DR-8 __	Range: 0 - 0.350 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 62		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Reagent Blank Correction: <input type="checkbox"/>
Notes: _____			
<b>Manganese (Mn<sup>2+</sup>):</b>			
Equipment	DR-850	DR-8 __	Range: 0 - 20.0 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 41		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Digestion: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	Reagent Blank Correction: <input type="checkbox"/>
Notes: _____			
<b>QA/QC Checklist:</b>			
All data fields have been completed as necessary: <input type="checkbox"/>			
Correct measurement units are cited in the SAMPLING DATA block: <input type="checkbox"/>			
Values cited in the SAMPLING DATA block are consistent with the Groundwater Sample Log Sheet: <input type="checkbox"/>			
Multiplication is correct for each Multiplier table: <input type="checkbox"/>			
Final calculated concentration is within the appropriate Range Used block: <input type="checkbox"/>			
Alkalinity Relationship is determined appropriately as per manufacturer (HACH) instructions: <input type="checkbox"/>			
QA/QC sample (e.g., Std. Additions, etc.) frequency is appropriate as per the project planning documents: <input type="checkbox"/>			
Nitrite Interference treatment was used for Nitrate test if Nitrite was detected: <input type="checkbox"/>			
Title block on each page of form is initialized by person who performed this QA/QC Checklist: <input type="checkbox"/>			



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
SOIL GAS SAMPLING

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## 1.0 PURPOSE

The purpose of this procedure is to provide general reference information on soil gas sampling. Soil gas investigations measure the general extent of volatile organic compound (VOC) contamination, such as chlorinated solvents and petroleum products, given off by subsurface soil and groundwater. The methods and equipment described are for collection of soil gas from the unsaturated zone of the subsurface soil.

## 2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for soil gas sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

## 3.0 GLOSSARY

Sampling Grid - Typically consists of a series of equal-distant sampling points set along parallel survey lines. The sample point spacing and number of parallel grid lines will depend upon the site specific conditions and project objectives.

Transect Line - A sampling network used to find the source area of contamination. Sampling points are placed along a transect line between the area of impact and a suspected source area. This can significantly decrease the number of points compared to a typical sampling grid.

Biased Location - Sample points are either placed near a suspected source area or in an anticipated clean area to refine the location of "hot spots" for further delineation or remediation.

Random Location - Random networks use a grid with numbers designating the nodes or areas within the grid. The sample points are then selected by a random number generator to designate which nodes or areas are targeted for sampling. This type of network is used in areas where little information is known or no contamination is suspected.

Combined Locations - This type of network is the most common used, and includes a combination of any of the four previous sampling networks mentioned.

Head Space Analysis - The screening or analysis of volatile organic vapors that have accumulated in the air space within a soil or groundwater sample container.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million (ppm) levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at ppm levels. A PID will not detect methane gas. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste (soil cuttings, purge water, etc.).

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#### 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate soil gas sampling procedure required to support the project objectives.

Field Operations Leader (FOL)-The FOL is primarily responsible for performing the soil gas sampling technique in accordance with the project-specific plan.

#### 5.0 PROCEDURES

##### 5.1 General

Soil gas methods are performed to delineate VOC contamination in subsurface soil and groundwater. Soil gas surveys are not intended to be a substitute for conventional subsurface methodologies (e.g., monitoring well installation and groundwater sampling) but rather are to be used as a screening technique to focus subsequent investigations to areas of potential concern. The advantages of using soil gas methodologies to define VOC contamination include:

1. Minimizing the number of subsequent test borings and monitoring wells required to characterize the nature and extent of the contamination.
2. Optimizing the placement of subsequent monitoring wells and test borings.
3. Allowing the collection of large amounts of data in a short time period relative to conventional methods.
4. Generating little or no investigative derived waste.

To assess the effectiveness of a soil gas survey, consider the following items:

1. The near surface geology.
2. The type of contamination present.
3. The anticipated concentration of contamination present.
4. The anticipated depth to the zone of contamination.

Common soil gas methods are most effective in geological settings that contain coarse textured soils (e.g., silty sands, sands and gravels) and are least effective in areas of fine textured soils (e.g., silty clays and clays). Soil gas methods may be effective at mapping areas of dense non-aqueous phase liquid VOC contamination that are present beneath the water table. Areas with deep water tables or low concentrations of VOC contamination may require the use of specialized soil gas techniques. These techniques could require the use of more expensive passive sorbent samplers left in place for a relatively long time period.

##### 5.2 Soil Gas Sampling Grids

The first stage of a soil gas survey is to establish a sampling grid or network. The grid should be designed to obtain all necessary information with a minimal expenditure of time and resources. The development of the grid should be based on background information regarding chemical properties of the contaminant, properties of the vadose zone, and hydrogeologic conditions of the area. All of this information should be used to design a sampling protocol specific to the conditions at the site. Some of the designs used include grids, transect lines, and biased, random or combined methodologies.

The size of the grid spacing is determined on a site specific basis. Locations should be marked with pin flags or wooden stakes and numbered sequentially at each site. The grid should be referenced to permanent site features and the stakes left in place to assist with future subsurface investigations. The location of any nearby surface or subsurface features which may affect the results of the survey (sewer

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line, fuel farm, etc.) shall be noted in the field notebook. Detailed notes and a sketch of each site, including station locations and station numbers will be documented in the field logbook. After the grid has been laid-out a dig permit must be applied for and approved prior to the start of sampling.

### **5.3 Sampling Methodologies**

There are several methods for the collection of soil gas samples. The most common types will be discussed in the following sections. Variations of the following methods may be conducted if approved by the Project Manager.

#### **5.3.1 Head Space Screening**

Head space screening is a method of obtaining rapid information concerning the presence of VOC contamination in the subsurface. Head space screening is usually applicable to soil samples but can also be modified to include groundwater samples. Although head space screening is not a direct measurement of insitu soil vapors, the procedure is included in this SOP because soil vapors are the actual media being screened or analyzed during headspace screening.

Upon sample retrieval from the subsurface, a small quantity of undisturbed soil (approximately 6 oz.) is removed from the sampler (e.g., hand auger, split-spoon sampler, Shelby tube, etc.) and immediately placed in a sealing (Ziploc®-type) plastic bag. Once sealed in the bag, the sample is gently massaged to break apart any large soil clumps. The bag is then warmed for 15 minutes in order to volatilize the potential contaminants from the soil sample into the head-space of the bag. For consistent readings at a particular site, it is important to warm each sample for the same time period and to approximately the same temperature. For this reason, placing the sample in the passenger compartment of a warm vehicle will provide a consistent ambient temperature for the procedure. Other methods of gently warming the sample can be used as long as the resulting temperature is consistent and the procedure is documented in the field log book. After 15 minutes, the tip of the PID or FID is carefully pushed directly through the plastic bag and a direct reading is obtained of the maximum detection. The PID or FID should be capable of storing the maximum detection value for a given reading. All head-space readings (maximum detection per sample) will be noted on the appropriate soil boring log and/or sample log form.

#### **5.3.2 Pipe Probes**

Pipe probes involve the use of a hollow steel tube to collect the soil gas sample. The probes can be passively placed into a predrilled hole or driven to the required depth. The predrilled hole can be made using a slide hammer, bucket auger, electric hammer, or DPT drill rig. The following three soil gas procedures using pipe probes are commonly used for preliminary screening in the field.

The first method described is the simplest and will provide immediate results that may help in the location of subsequent test pits, soil borings, etc. The steps are as described below:

- Drive a 3/4-inch steel pipe into the ground using a slide hammer, electric hammer or equivalent to the desired depth. Generally this will not exceed five feet due to the difficulty in retrieving the pipe.
- Remove the steel pipe from the ground by hand or jack.
- Place a hollow 3/4-inch diameter by 1 foot long steel pipe into the previously created hole and seal off the outside of the pipe with bentonite clay so that no soil gas can escape. Allow 5 minutes for vapors in the hole to reach equilibrium conditions.
- Attach the tip of the PID/FID to the hollow pipe using silicon tubing and collect instrument readings for a minimum of 1 minute, or until the readings peak and begin to decline. Record the highest value in the field notebook along with the time, date, and sample location.

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- Decontaminate the steel pipes before moving to the next location to minimize the potential for cross-borehole contamination.

Note: Any ancillary observations made such as soil type, soil color, depth to water table, etc., should be recorded in the field logbook.

The next method is a variation of the first, but provides a more depth specific sample and uses a Tedlar® bag for the collection of the soil gas.

- Drive an expandable steel drive point (3/4-inches diameter) attached to a 5 foot connecting rod into the ground using a slide or electric hammer or DPT drill rig.
- Retract the rod a sufficient distance to leave a space for the soil gas to enter the drive point (approximately 6 inches). Allow 5 minutes for vapors in the hole to reach equilibrium conditions.
- After the drive point and connecting rod have been retracted, assemble the sampling apparatus by attaching 1/4-inch I.D. polyethylene tubing to the top of the connecting rod.
- Place the tubing to the inlet side of a peristaltic pump and connect the discharge side of the pump to a 1-liter Tedlar® bag.
- Fill the Tedlar® bag twice to adequately purge the sample equipment and ambient air that exists in the bag.
- Collect the soil gas sample in a separate Tedlar® bag as above and then connect it directly to the inlet of a PID/FID.
- Obtain the maximum reading from the PID/FID in ppm and record the data in the field notebook or sample log form.
- Decontaminate the equipment before moving to the next location.

The third procedure is similar to the above, except that the soil gas collected in the Tedlar® bag is analyzed in the field using either a portable or onsite lab gas chromatograph (GC). This procedure can identify specific contaminant(s) of concern. These instruments, though more expensive, can be very sensitive and selective to the contaminant(s) of concern.

### 5.3.3 Passive Sorbent Samplers

The most common type of sorbent sampler used is known as a Petrex® tube, which consists of activated charcoal chemically fused to the tip of a Curie-point ferromagnetic wire and inserted into a glass tube. The collector is then buried at a depth of 2 to 4 feet in an inverted position with the glass tube acting as a flux chamber for an optimal period of time as determined by the manufacturers recommendations. Sample analysis is by thermal desorption onto a mass spectrometer (MS) or GC.

This method is recommended when the contaminants are unknown and concentrations are expected to be low. Specialized sorbent samplers may require the use of a soil gas subcontractor.

### 5.3.4 Well Points

Well points can be installed to obtain data on subsurface gas concentrations at depths or areas inaccessible by other monitoring techniques. Single or multiple probes may be installed in a single

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borehole. Well points are recommended for projects if more than one soil gas sampling event is to occur to monitor contaminant migration versus time. The construction of the well points may vary and may require the use of a conventional drill rig, DPT drill rig, power auger, or hand auger. The need for this type of soil gas survey shall be determined by the Project Manager and site specific conditions. The installation of a soil gas well point is described below:

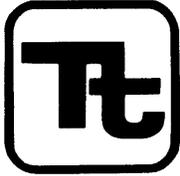
- Drill the borehole to the desired depth.
- Set the screened well point at the desired depth and backfill with a clean silica sand to approximately 1 foot above the screened length.
- Place soil cuttings or bentonite pellets on top of the sand pack.
- Place 1 to 2 feet of a cement/bentonite grout on top of the bentonite plug.
- If necessary, install a steel protective casing over the well point to prevent damage.
- Place sample outlets inside the protective casing for sampling.

**6.0 REFERENCES**

New Jersey Department of Environmental Protection and Energy, Field Sampling Procedures Manual, May, 1992.

**7.0 RECORDS**

A record of all field procedures, tests, and observations must be recorded in the field logbook. Entries should include all pertinent data regarding the soil gas survey. The use of sketches, photographs, and field landmarks will help to supplement the investigation and evaluation.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject  
FIELD DOCUMENTATION

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

## 2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

## 5.0 PROCEDURES

### 5.1 SITE LOGBOOK

#### 5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

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- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

### **5.1.2 Photographs**

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

## **5.2 FIELD NOTEBOOKS**

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

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separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

### **5.3 FIELD FORMS**

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (<http://intranet.ttnus.com>) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

#### **5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results**

##### **5.3.1.1 Sample Log Sheet**

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

##### **5.3.1.2 Sample Label**

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

##### **5.3.1.3 Chain-of-Custody Record**

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc<sup>®</sup>-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

##### **5.3.1.4 Chain-of-Custody Seal**

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

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and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

#### 5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

### 5.3.2 **Hydrogeological and Geotechnical Forms**

#### 5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

#### 5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

#### 5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

#### 5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

#### 5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

#### 5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

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### 5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

### 5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

### 5.3.3 **Equipment Calibration and Maintenance Form**

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

## 5.4 **FIELD REPORTS**

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

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#### **5.4.1 Daily Activities Report**

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

##### **5.4.1.1 Description**

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

##### **5.4.1.2 Responsibilities**

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

##### **5.4.1.3 Submittal and Approval**

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

#### **5.4.2 Weekly Status Reports**

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at <http://intranet.ttnus.com> under Field Log Sheets.

#### **6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE**

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

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- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

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**ATTACHMENT A  
TYPICAL SITE LOGBOOK ENTRY**

START TIME: \_\_\_\_\_ DATE: \_\_\_\_\_

SITE LEADER: \_\_\_\_\_

PERSONNEL: \_\_\_\_\_

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well \_\_\_\_ resumes. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well \_\_\_\_\_.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well \_\_\_\_\_.
4. Well \_\_\_\_\_ drilled. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 2, page \_\_\_\_ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well \_\_\_\_\_ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manager arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit \_\_\_\_\_.
8. Test pit \_\_\_\_\_ dug with cuttings placed in dump truck. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit \_\_\_\_ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

\_\_\_\_\_  
Field Operations Leader

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**ATTACHMENT B  
SAMPLE LABEL**

	Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
			Site:
		Location:	
Sample No:		Matrix:	
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	



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**ATTACHMENT D  
CHAIN-OF-CUSTODY SEAL**

<u>Signature</u> <hr/> <u>Date</u> <hr/> <b>CUSTODY SEAL</b>		<b>CUSTODY SEAL</b> <hr/> <u>Date</u> <hr/> <u>Signature</u>
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# STANDARD OPERATING PROCEDURES

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Effective Date 01/28/2009	Revision 6
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

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## 1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment, chemical sampling equipment and field operation and analytical equipment.

## 2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

## 3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent - A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

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#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

#### 5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

#### 6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

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- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

## 7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

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- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

## 7.1 Decontamination Pad Design/Construction Considerations

### 7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:
  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

The selected decontamination site should include the following, where possible:

- Areas where potable water and electricity are provided.

#### **Safety Reminder**

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).
- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

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- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.
- Areas that offer sufficient size to carry out the specific decontamination sequence.
- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:
  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.
  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.
  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.
  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.
  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:
  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

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- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.
- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

### **7.1.2 Decontamination Activities at Drill Rigs/DPT Units**

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

### **7.1.3 Decontamination Activities at Remote Sample Locations**

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

## **7.2 Equipment Decontamination Procedures**

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

### **7.2.1 Monitoring Well Sampling Equipment**

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.
2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.
3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.
4. Remove the pump and tubing/bailer from the container
5. Rinse external pump components using tap water.

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6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

**CAUTION**

Do not rinse PE, PVC, and associated tubing with solvents –  
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.
8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.
9. Drain residual deionized water to the extent possible.
10. Allow components of the equipment to air dry.
11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.
12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

**SAFETY REMINDER**

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

**NOTE**

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

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### 7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.
- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:
  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

### 7.2.2 **Downhole Drilling Equipment**

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

**CAUTION**  
 Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

**CAUTION**  
 In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

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4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol
5. To the extent possible, allow components to air dry.
6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.
7. Wrap or cover equipment in clear plastic until it is time to be used.

**SAFETY REMINDER**

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.
2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.
3. Always wear PPE as specified in the HASP such as:
  - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.
4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.
5. Do not modify equipment unless the manufacturer has approved the modifications.

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### 7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

**CAUTION**

Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

**CAUTION**

When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

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### 7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

#### 7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

**NOTE**

Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

**NOTE**

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).
4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:
  - Enclose areas accessible by the general public using construction fencing and signs.
  - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
  - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
  - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
  - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
  - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

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- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

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**CAUTION**

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

**7.4 Decontamination Evaluation**

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.
- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

**NOTE**

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:
  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.
  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

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**NOTE**

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.



# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject DECONTAMINATION OF FIELD EQUIPMENT

## Attachment A iDW Label

**INVESTIGATION DERIVED WASTE**

GENERATOR INFORMATION:

SITE \_\_\_\_\_ JOB NO. \_\_\_\_\_

LOCATION \_\_\_\_\_

DATE \_\_\_\_\_

DRUM# \_\_\_\_\_

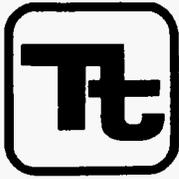
CONTENTS \_\_\_\_\_

VOLUME \_\_\_\_\_

CONTACT \_\_\_\_\_

EMERGENCY PHONE NUMBER \_\_\_\_\_





TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Effective Date	09/03	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>DS</i>		

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)

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## 1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

## 2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

## 3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

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Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

## **5.0 SOIL SAMPLING PROCEDURES**

### **5.1 General**

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

### **5.2 Sampling Equipment**

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

### **5.3 DPT Sampling Methodology**

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.
- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.
- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.
- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.

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- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

## 6.0 GROUNDWATER SAMPLING PROCEDURES

### 6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

### 6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

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### 6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.
- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.
- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ( $\pm 10$  percent), the well may be sampled.
- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.
- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).
- Decontaminate the equipment before moving to the next location.

### 7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

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**ATTACHMENT 1  
SAFE WORK PERMIT FOR DPT OPERATIONS**

Permit No. \_\_\_\_\_ Date: \_\_\_\_\_ Time: From \_\_\_\_\_ to \_\_\_\_\_

**SECTION I: General Job Scope**

- I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**
- II. Required Monitoring Instruments: \_\_\_\_\_
- III. Field Crew: \_\_\_\_\_
- IV. On-site Inspection conducted  Yes  No Initials of Inspector TtNUS

**SECTION II: General Safety Requirements (To be filled in by permit issuer)**

- V. Protective equipment required
  - Level D  Level B
  - Level C  Level A
  - Detailed on Reverse
- Respiratory equipment required
  - Full face APR
  - Half face APR
  - SKA-PAC SAR
  - Skid Rig
- Escape Pack
- SCBA
- Bottle Trailer
- None

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern	Action Level(s)	Response Measures
_____	_____	_____

VII. Additional Safety Equipment/Procedures

- |   |  |
|---|--|
| Hard-hat ..... <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No                | Hearing Protection (Plugs/Muffs) <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
| Safety Glasses ..... <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No          | Safety belt/harness <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No              |
| Chemical/splash goggles ..... <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No | Radio <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No                            |
| Splash Shield ..... <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No           | Barricades <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No                       |
| Splash suits/coveralls ..... <input type="checkbox"/> Yes <input type="checkbox"/> No             | Gloves (Type - _____) <input type="checkbox"/> Yes <input type="checkbox"/> No                       |
| Steel toe Work shoes or boots <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Work/warming regimen <input type="checkbox"/> Yes <input type="checkbox"/> No                        |

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors	Yes	NA	Yes	NA
Safety shower/eyewash (Location & Use).....	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Daily tail gate meetings.....	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Contractor tools/equipment/PPE inspected.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Emergency alarms.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Evacuation routes.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Assembly points.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

IX. Site Preparation

- Utility Clearances obtained for areas of subsurface investigation  Yes  No
- Physical hazards removed or blockaded  Yes  No
- Site control boundaries demarcated/signage  Yes  No

X. Equipment Preparation

- |  |                              |  |
|--|------------------------------|--|
| Equipment drained/depressurized.....                       | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Equipment purged/cleaned.....                              | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Isolation checklist completed.....                         | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Electrical lockout required/field switch tested.....       | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Blinds/misalignments/blocks & bleeds in place.....         | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Hazardous materials on walls/behind liners considered..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |

- XI. Additional Permits required (Hot work, confined space entry).  Yes  No  
*If yes, complete permit required or contact Health Sciences, Pittsburgh Office*

XII. Special instructions, precautions:

\_\_\_\_\_

\_\_\_\_\_

Permit Issued by: \_\_\_\_\_ Permit Accepted by: \_\_\_\_\_

**APPENDIX E**

**ANALYTICAL LABORATORY SOPs**

A.P.P.L., INC.  
CONFIDENTIALQA CONTROL COPY # 4**Standard Operating Procedure****High Resolution GC-Mass Spec Periodic Maintenance SOP****STATEMENT OF PURPOSE**

This procedure will include methods for performing periodic maintenance of GC inlet and column systems. High Resolution Mass Spec maintenance is performed by the manufacturer.

**INSTRUCTIONS**

For all GC maintenance procedures turn the oven temperature down to 20°C and shut off gas flows.

**A. Routine GC maintenance:**

- 1) At least once a week or for every 100 injections, the capillary inlet inserts will be replaced. The septa will be checked at least every 100 injections or more often as necessary.
  - a) Chromatograms exhibiting peak tailing, low peak response of standards or breakdown products may indicate that immediate maintenance is needed.
  - b) Allow the injection port to cool to room temperature and shut off the gas flows to the instrument. Remove the septa nut and replace the used septa with a new one. Replace the nut with hand tight plus ¼ turn of a wrench.
  - c) Remove the injection port assembly using a wrench to loosen the nut. Take out the used glass inlet liner and replace it with a new one.
  - d) Bake the GC for one hour using the following temperatures: Oven 290°C, injector ports 275°C, and detectors 350°C. Column maximum temperatures are listed in the documentation accompanying a new column.
  - e) A continuing calibration check standard will be injected to verify the calibration curve. If continuing calibration fails to meet criteria as stated in the method, a complete linearity of standards applicable to each type of analysis will be injected.

**B. Periodic GC maintenance:**

The following maintenance will be performed when degradation of standards indicates a dirty split seal:

- 1) Replacing split seal (frit) for capillary inlet systems:
  - a) The split seal (frit) (fig. 18-3 HP ref. Manual II) is commonly contaminated when dirty samples are run through the GC. Reduced peak size or breakdown peaks may indicate cleaning of the split seal is needed.

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CONFIDENTIAL

QA CONTROL COPY # 4

- b) After turning GC temperatures down, remove the column from the injection port and unscrew the reducing nut.
- c) The split seal (frit) and flat washer located inside the reducing nut are removed and replaced.
- d) Re-install column and turn the oven temperature up to 290°C until the detector signal is stable.

The following maintenance will be performed when chromatography and trouble shooting indicates a dirty injector port.

## 2) Cleaning injector ports:

The following maintenance will be performed when chromatography and trouble shooting indicates a dirty injector port.

- a) Turn off the oven and remove the analytical column when the oven has cooled.
- b) Lower the injection port temperature to room temperature.
- c) Remove the glass injection port insert.
- d) Place a beaker beneath the injector port inside the GC oven. Using a Teflon wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene, catching the rinsate in the beaker.
- e) Prepare a solution of deactivation agent (Sylon-CT or equivalent) following manufacturer's directions. Coat all metal surfaces inside the injector body with the deactivation solution, then serially rinse the injector body with toluene, methanol, acetone and hexane.
- f) Reassemble the injector and replace the GC column.

## 3) Cutting the Column:

- a) With continued sample analysis, contaminants tend to build up in the injection port side of the capillary column, causing decreased peak response and peak tailing.
- b) Using a wrench, remove the column inlet nut from inside the GC oven. Pull the column out of the nut and remove the graphite ferrule within the nut.
- c) Use a column scoring tool to remove 1 –2 loops from the inlet side of the column and discard.
- d) Thread the inlet nut back onto the column, and then slide a new ferrule onto the column. Allow the column to protrude 6mm from the top of the ferrule and replace back into the inlet using a wrench to tighten.

A.P.P.L., INC.  
CONFIDENTIALQA CONTROL COPY # 4**Annual Gas Line Purifier Replacement**

- 1) Changing oxygen/moisture indicator tube (OMI-1): The OMI-1 tube, located downstream from the gas purifier, indicates potential contamination within the carrier gas by rate of color change in the tube.
  - a) Turn down GC temperatures.
  - b) Unplug the heated converter tube. Turn off gas flow to line.
  - c) Using the parts in the OMI-1 installation kit, slide end caps onto ends of gas line, then finger tighten reducing union bodies to line ends. Wrench tighten approximately 1¼ additional turns.
  - d) Carefully remove plastic end caps from new tube. Avoid damaging foil seals that protect tube contents from exposure to air. Seals will be punctured by piercing needles in reducing unions when the tube is connected to the system.
  - e) Place nuts and new ferrules on the tube. Push exit of the tube into the reducing body closest to the instrument. Screw the nut and ferrule onto the union body and finger tighten. The piercing needle will penetrate the foil seal as you push the tube into the union. Slide the tube holder over the OMI-1 tube and gas line until the tube inlet is exposed.
  - f) Push the inlet end of the tube into the reducing union body. Position a slotted washer for the tube holder over the junction in one reducing union. Place the end cap on the tube holder and tighten until snug. Repeat at other end of tube holder. Turn the gas back on.
  - g) DO NOT ATTEMPT TO REMOVE THE CONTENTS OR REUSE THE TUBE. Spent resin contains a strong alkali.

Any partially spent tube should be placed in a glass beaker and stored away from combustible materials until the resin is a uniform brown. Dispose of the tube as a hazardous solid waste in accordance with applicable federal, state and local regulations.

**D. HR Mass Spec Maintenance**

- 1) APPL Inc. maintains a service contract with the instrument manufacturer (Waters Inc). Due to the technical nature of the HRMS, only Waters Inc service engineers are allowed to perform maintenance on the following mass spec components: the source, the flight tube and the mass spec detector.
- 2) Place a service call to Waters Inc if the following are observed: decrease in detector response, problems calibrating the mass axis, decreased beam transmission, low resolution checks or standard calibration problems with the ICAL or CCVs.



Standard Operating Procedure

A.P.P.L., INC.

SOP: 8290MAIN  
Section: 10  
Revision: 0  
Date: 11/19/08

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SAFETY

QA CONTROL COPY # 4

This procedure is applicable to HRGCMS instrumentation personnel who have been allowed to perform period maintenance.

Section Manager: Maandehulew

Date: 11/19/08

QAU Director: Francis Lohman

Date: 11/19/08



# Standard Operating Procedure

## Instrumental Analysis of Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) BY HRGC-HRMS (EPA METHOD 8290<sup>1</sup>)

### STATEMENT OF PURPOSE

This procedure describes the part per trillion instrumental analysis of PCDD (Dioxins) and PCDF (Furans) in extracts from solid, tissue and aqueous matrices using high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

### INSTRUCTIONS

#### **1.0 Scope and Application**

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. If an individual project has its own QAPP with client specific requirements that are different than the SOP, the QAPP overrides the SOP. This information will be specified in the comment section of the ARF.

- 1.1 The quantitation range of 2,3,7,8-TCDD for 1 liter water sample (final vol. 50 $\mu$ L) is 0.02 – 4.0ppt (ng/L). The quantitation range of 2,3,7,8-TCDD for 10g soil/tissue (final vol. 50 $\mu$ L) is 2.0 – 400ppt (ng/kg). Reporting limits are based upon the lowest calibration point and the dilution factor, however Practical Quantitation Limits (PQL) are dependent on the potential interferences caused by the sample matrix.
- 1.2 The Toxicity Equivalent (TEQ<sub>pcb</sub>) using 2,3,7,8-TCDD may also be determined by this method.
- 1.3 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 extracted cleaned up according to APPL SOPs SEP8290 (aqueous), SOX8290S (soils/sediment), and SOX8290F (fish tissue/paper pulp).
- 2.2 C13 labeled internal standards are added to each sample extract prior to instrument analysis. The analytes are separated by HRGC using a Restek DB-5 column and detected by HRMS.
- 2.3 An analyte is identified in a sample by comparing the RT and ion abundance ratios (of the two most abundant m/z) to the standard RT and theoretical ion abundance ratios.
- 2.4 Quantitation is achieved based on a five-point calibration curve, using the internal standard quantitation technique.

#### **3.0 Sample Preservation, Containers, Handling and Storage**

- 3.1 Containers used to collect samples for the determination of Dioxin and Furan compounds are purchased pre-cleaned. The sample containers for solids are wide mouth, amber glass, 500mL minimum containers with Teflon lined screw caps. The sample containers for waters are 1 liter amber glass bottles with Teflon lined screw caps. Filleted fish tissue samples may be collected in aluminum foil and kept frozen until receipt by the laboratory.
- 3.2 All samples will be taken and held at a temperature of 4°C  $\pm$  2°C in the dark until delivery



to the laboratory. Water and solid samples are then placed into a refrigerator that is kept at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  until extraction. Tissue samples are to be stored frozen by the laboratory until extraction.

- 3.3 Extraction hold time is 30 days from date of collection, and analysis hold time is 45 days from date of collection.

#### 4.0 Interferences and Potential Problems

- 4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts, elevated baselines, and/or lock-mass suppression causing misinterpretation of chromatograms. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Where possible, reagents are cleaned by extraction or solvent rinse. Baking of glassware in a kiln or furnace at  $450 - 500^{\circ}\text{C}$  may be necessary to remove contaminants.
- 4.2 All materials used in the analysis must be demonstrated to be free from interferences by running reference matrix method blanks initially and with each sample batch.
- 4.3 Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the target analytes. The most frequently encountered interferences are polychlorinated biphenyls. Because very low levels of Dioxins and Furans are measured by this method, the elimination of interferences is essential. The cleanup steps given in the extraction SOPs can be used to reduce or eliminate these interferences and thereby permit reliable determination of the analytes at low levels.
- 4.4 In order to prevent contamination of the calibration solutions, the solutions must be prepared in an area free from contamination using glassware free from contamination.
- 4.5 If the laboratory air is a potential source of contamination, samples, reagents, glassware, and other materials should be dried in a glove box or other area free from contamination.

#### 5.0 Equipment/Apparatus

- 5.1 Laboratory Fume Hood certified for radioactive compounds.
- 5.2 Gas chromatograph—Must have splitless or on-column injection port for capillary column, temperature program with isothermal hold, and must meet all of the performance specifications in Section 7 of this SOP.
- 5.3 GC column—
- 5.3.1 The suggested primary column is 60M DB-5, and the suggested confirmation column (for resolving 2,3,7,8-TCDF from the individual isomers) is 30M DB-225. If the isomers listed in the method (2,3,4,7-TCDF and 1,2,3,9-TCDF) are resolved by 25% on the DB-5 column, then the confirmation column analysis is not necessary for 2,3,7,8-TCDF.
- 5.3.2 The column must meet the specifications listed in this SOP for retention time and resolution of peaks.
- 5.2 Mass spectrometer—28- to 40-eV electron impact ionization, must be capable of selectively monitoring a minimum of 22 exact  $m/z$  minimum at high resolution (10,000) during a period less than 1.5 seconds.
- 5.3 GC/MS interface—The mass spectrometer (MS) must be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beams.
- 5.4 Data system—Capable of collecting, recording, storing, and processing MS data



(MassLynx Software). Data acquisition—The signal at each exact m/z must be collected repetitively throughout the monitoring period and stored on a mass storage device.

- 5.5 Class A volumetric flask - 10mL and 25mL for preparation of standards.
- 5.6 Class A volumetric syringes-10 $\mu$ L, 100 $\mu$ L, 500 $\mu$ L, and 1.0mL for standard and spike preparation.

## 6.0 Reagents

- 6.1 Reagent or pesticide grade chemicals shall be used in all tests. 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. All reagents and chemicals will be documented properly for traceability. All solvents will be pesticide quality and each lot of solvent.
- 6.2 NOTE: Store the standard solutions (stock, calibration and internal standards) at 4°C in Teflon-sealed containers in the dark. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem. It is recommended that the laboratory purchase dilute standard solutions of the analytes in this Method. However, if primary solutions are prepared, they must be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator must be worn when high concentrations are handled.
- 6.3 Organic-free reagent water: All references to water in this method refer to organic-free reagent water.
- 6.4 Stock standard solutions may be purchased as individual manufacturer certified solutions. Stock solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Reference standards that can be used to determine the accuracy of standard solutions are available from several vendors. The certified solutions must be accompanied by a certificate of analysis that states 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. Standards formulated in house are prepared with balances that are calibrated with weights traceable to NIST. (See SOP ROU003)

## 6.5 Dioxin and Furan Analytical Standards

- 6.5.1 Calibration Standards- may be purchased from a manufacturer such as Cambridge Isotope in five separate solutions in nonane, as listed in Table 1 of this SOP. The calibration standards should include the seventeen unlabeled Dioxins and Furans, as well as the nine C13-labeled internal standards and the two C13-labeled recovery standards. The CS-3 standard is used for calibration verification (CCV).
- 6.5.2 Performance check solution- may be purchased in nonane solvent from a manufacturer such as Cambridge Isotope to include the compounds listed in Table 2 of this SOP.
- 6.5.3 Internal Standards- may be purchased from a manufacturer such as Cambridge Isotope to include the compounds listed in Table 3 of this SOP. This mix is prepared in nonane and added to each sample, blank and spike prior to extraction.



# Standard Operating Procedure

## QA Control Copy # 3

SOP: HPL8290  
Section: 10  
Revision: 4  
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- 6.5.4 Recovery Standards- may be purchased from a manufacturer such as Cambridge Isotope to include the compounds listed in Table 3 of this SOP. This mix is prepared in nonane and added to each sample, blank and spike at the final concentration step of the extraction procedure.
- 6.5.5 Spike Mix- may be purchased from a manufacturer such as Cambridge Isotope to include the compounds listed in Table 4 of this SOP. This mix is prepared in nonane and added to each LCS and MS/ MSD prior to extraction.

### 7.0 Procedure

Extraction: Refer to APPL, Inc.'s SOPs for the following extraction procedures:

- 7.1 Water:
  - SOP# SEP001- Proper Use of Separatory Funnels (EPA Method = NA)
  - SOP# SEP8290 – Dioxin/Furan Separatory Funnel Extraction (EPA Method 8290)
- 7.2 Soil/Sediment:
  - SOP# SOX001- Set Up and Operation of Soxhlet Extraction Apparatus (EPA Method = NA)
  - SOP# SOX8290S - Dioxin/Furan by Soxhlet Extraction (EPA Method 8290)
- 7.3 Fish Tissue/Paper Pulp:
  - SOP# SOX001- Set Up and Operation of Soxhlet Extraction Apparatus (EPA Method = NA)
  - SOP# SOX8290F - Dioxin/Furan by Soxhlet Extraction (EPA Method 8290)
- 7.4 Extract cleanup:
  - SOP # SEP8290, SOX8290S, SOX8290F
  - The extract will be transferred to an injection vial with Teflon-lined cap. Proceed with gas chromatographic analysis if further cleanup is not required.
- 7.5 Recommended Operating Conditions for HRGC:
  - Column: Carrier gas (He) flow rate: 1 mL/min.
  - Oven Temperature: 200°C
  - Equilibration Time: 1.0min
  - Initial Temp: 200°C
  - Initial Time: 2min
  - Temp Ramp: 5 °C/min to 220°C and hold 16 min, ramp 5°C/min to 235°C and hold 7 min, then ramp 5°C/min to 330°C and hold 5 min.
  - Total Run Time: 60 min
  - Injector Temp: 270°C
  - Interface Temp: 290°
  - Injection Volume: 1µL
- 7.6 These conditions may be used as guidelines to establish an optimal GC temperature program. With the possible coelution of sample components, it may be necessary to adjust chromatographic conditions to give adequate separation of the characteristic peaks between individual congener peaks. Once a temperature program has been established, all samples must be analyzed under the same operating conditions as standards.
- 7.7 The HRGC run sequence should be arranged in the following manner:
  - GC Column Performance Check Solution
  - CS-0.2



CS-1  
CS-2  
CS-3  
CS-4  
CS-5  
Nonane Blank  
GC Column Performance Check Solution  
CS-3 (CCV)  
12 hour analytical shift (including blanks, spikes and samples)  
CS-3 (CCV)

### 7.8 Instrument Calibration for Quantitative Analysis

- 7.8.1 One of the concentrations will be at the quantitation limit. The analyst must refer to the incoming sample notice for the lab works code and look at the detection limits listed on the appropriate form 1 to determine the quantitation limit standard. The initial calibration curve is a reflection of the performance of the instrument at any given time. Individual compounds react to the changing dynamic of the instrument. Therefore it is sometimes necessary to delete points for individual compounds in an initial calibration curve. When this occurs the following rules are followed to ensure integrity of the data:
- 7.8.2 A standard must be included in the curve for each compound, which is less than or equal to the reporting limit. If the responses of a sample peak exceed the calibration range of the system, dilute the extract and reanalyze.
- 7.8.3 The deletion of discrete points must never result in a calibration curve consisting of less than five points for each analyte of interest.
- 7.8.4 Points for an individual analyte in the middle of the curve may not be deleted, however unforeseen circumstances may occur such as a miss injection by the autosampler, a loose cap on an injection vial, etc. In this situation the entire level is deleted for all compounds and the reason for deletion is noted on the multilevel form. If this results in a calibration curve that consists of less than five points, another level may be run before the analysis of samples begin.
- 7.8.5 Points at the low end and high end of the curve may be deleted if it is determined the compound ceases to be linear at either end. Any positive findings in the samples will be analyzed so as to fall within the linear range of that particular compound.
- 7.8.6 Tuning with PFK – Tuning conditions on the groups of monitored ions shown in Table X.
- 7.8.7 Initial Calibration (ICAL) Analyze the five calibration standards (see Table 1) by HRGC. Record the sum of the peak areas for each of the two m/z of interest for each congener. The results can be used to prepare a calibration curve for each analyte. The ratio of the response to the amount injected, defined as the response factor (RF), can be calculated for each analyte at each standard concentration. If the percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average response factor can be used in place of a calibration curve. When this criterion is exceeded, inspect the HRGC system to determine the cause and perform whatever maintenance is necessary before re-calibrating and proceeding with analysis. The following criteria must also be met



for the ICAL in order for sample analysis to proceed, otherwise the mass spec will need to be adjusted and the ICAL repeated.

- 7.8.7.1 The signal to noise ratio for each native and C13 labeled standard must be >10.
- 7.8.7.2 The ion abundance ratios must be within acceptance method criteria (See Table 5).
- 7.8.7.3 The %RSD for native compounds is <20%, and the %RSD for C13 labeled compounds is <30%.

7.8.7.4 Internal Standard Calibration Technique is used to determine PCDD/PCDF C13 surrogates.

7.8.7.4.1 Calibration is achieved using the average response factors of the fixed concentrations of C13 surrogates in each of the points of the calibration curve (See Table 1). The nearest-eluted C13 I.S. is used for quantitation for each C13 surrogate. The %RSD for the labeled standards Average RF should be 30% or lower.

7.8.7.4.2 The peak areas of the internal standards for a particular sample, blank or spike must be +/- 30%D compared to the average RF of the I.S. in the ICAL.

7.8.7.4.3 Response factors are calculated as follows:

$$\text{Response Factor (RF)} = \frac{A_s C_{is}}{A_{is} C_s}$$

Where:

- $A_s$  = Sum areas of both m/z for the C13 surrogate
- $A_{is}$  = Sum areas of both m/z for the IS
- $C_s$  = Concentration of calibration standard
- $C_{is}$  = Concentration of IS

7.8.7.5 Isotope Dilution Technique is used to determine the native PCDD/PCDF compounds.

7.8.7.5.1 Calibration is achieved using the average response factors of the increasing concentrations of native PCDD/PCDF in the initial calibration curve. The corresponding C13 surrogate is used to quantitate each native compound in the samples and spikes. The %RSD for the native standards average RF should be 20% or lower.

7.8.7.5.2 Response factors are calculated as follows:

$$\text{Relative Response Factor (RR)} = \frac{A_n C_L}{A_L C_n}$$

Where:

- $A_n$  = Sum areas of both m/z for the native PCDD/PCDF
- $A_L$  = Sum areas of both m/z for the C13 PCDD/PCDF
- $C_n$  = Concentration of native calibration standard
- $C_L$  = Concentration of C13 calibration standard.

The % RSD is calculated as follows:



$$\%RSD = (SD)(100\%)/(RF_{x1})$$

Where:

- %RSD = Percent relative standard deviation.
- RF<sub>x1</sub> = Mean of the initial RR for a compound.
- SD = Standard deviation of the average RRF for a compound

- 7.9 Continuing Calibration Verifications (CCVs) must be analyzed at the beginning and end of each 12-hour analytical shift, by injecting the CS-3 calibration standard. The following CCV acceptance criteria must be met in order for analysis to proceed, otherwise the mass spec must be adjusted and a fresh CCV prepared and analyzed until the acceptance criteria can be met. If the mass spec adjustment includes changing the resolution, then a new resolution check must also be prepared and analyzed. If these corrective actions are not successful, then a new ICAL must be prepared and analyzed.
- 7.9.1 The ion abundance ratios must be within acceptance method criteria (See Table 5).
  - 7.9.2 For the beginning CCV, the %D of the native PCDD/PCDF should be within 20% from the average RF from the ICAL. The %D of the C13 labeled compounds should be within 30% from the average RF from the ICAL.
  - 7.9.3 For the ending CCV, the %D of the native PCDD/PCDF should be within 25% from the average RF from the ICAL. The %D of the C13 labeled compounds should be within 35% from the average RF from the ICAL.

$$\%D = A - B / A$$

- D = Difference
- A = Avg RF from the ICAL
- B = RF from the CCV

NOTE: The method allows for marginal %D failures in certain cases, such as the C13 labeled compound has a %D outside control limits while the corresponding native compound is acceptable.

- 7.10 Each sample analysis sequence must include an acceptable initial calibration; calibration verification standards and resolution check standards. When a CCV or Resolution Check fails to meet the acceptance criteria, all samples that were injected after the last acceptable check must be re-injected.
- 7.11 Sample injection may continue for as long as all the calibration verification standard requirements listed above are met.
- 7.12 Mass Spec Resolution
  - 7.12.1 Using Perfluorokerosene (PFK) and a molecular leak, tune the instrument to meet resolution 10,000 (10% valley) at high reference m/z 380.9760 and low reference m/z 304.9824.
  - 7.12.2 PFK is used to correct for mass-drift that may occur during the long analysis time. The lock-mass is established from PFK and is dependent on the m/z monitored within each descriptor. The deviation between the m/z values listed in the method and the actual m/z values must be less than 5ppm.



- 7.12.3 Obtain a selected ion current profile (SICP) at the two  $m/z$  specified in the EPA method 8290<sup>1</sup>, Table 6 at 10,000 resolution for each LOC for all native and labeled PCDD/PCDF. The resolution throughout the mass range for each function must be 8000; however the resolution in the center of the mass range for each function must be 10,000.
- 7.12.4 Resolution checks must be analyzed at the beginning and end of each analytical shift or at least every 12 hours. The MassLynx software includes a feature called "Experimental Calibration" that meets the resolution check method requirement. If the MS resolution criteria listed above can not be met, then analysis may not proceed. Samples affected by poor closing resolution check must be re-analyzed.
- 7.13 GC Performance Check
- 7.13.1 At the beginning of each 12 hour analytical shift a performance check standard will be run. See table 2. The peak to valley resolution for the tetra dioxin must be <25% of the highest peak in the chromatogram.
- 7.14 Qualitative Determination: A native or labeled congener is identified in a standard, blank or sample when all of the following criteria are met:
- 7.14.1 The signals for the two  $m/z$  listed in the EPA method 1668<sup>1</sup>, Table 7 must be present and must maximize within the same two scans. The area ratio of the two  $m/z$  must be within the criteria listed in Table 5 of this SOP for positive identification to be determined.
- 7.14.2 The S/N ratio at the  $m/z$  of interest must be greater or equal to 2.5 for each compound detected in the sample extract and greater or equal to 10 for all congeners in the ICAL and CCV standards.
- 7.14.3 Retention times are crucial to the identification of target congener compounds. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. The RT of a native compound must be within (-1 to +3) seconds of the corresponding C13 compound. If the native compound does not have a corresponding C13 compound (as shown in Table 1), then the RT of the native must fall within 0.005 units of the relative RT measured in the nearest CCV.
- 7.14.4 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their retention time windows, the system is out of control. Determine the cause of the problem and correct it.
- 7.15 Homolog Totals by LOC Estimation: The total concentration of all PCDD/PCDF's at a given Level of Chlorination may be reported by summing the concentrations of all congeners detected at that LOC.
- 7.16 Toxicity Equivalent Concentration (TEQ): The TEQ for a particular environmental sample may be determined by summing the concentrations of each individual toxic PCDD/PCDF multiplied by their respective Toxicity Equivalent Factors (TEF) as described in the EPA method 8290<sup>1</sup>.
- 7.17 Manual integration  
Make a hard copy of the original integration performed by the instrument. Perform the appropriate manual integration. Make a copy of the manual integration. Be sure that the scale of the copy is appropriate. Note on the manual integration copy the number of the reason (see list below) a new integration was performed, initial and date. Save the manual integration electronically so that it can be retrieved at a later date if necessary.



Place a copy of all integrations in the data folder to be reviewed by the section manager or his/her designee.

- (1) Integration does not follow baseline
- (2) Non-target peak interference
- (3) To split a peak
- (4) To integrate a split peak
- (5) The peak was not integrated
- (6) Computer integrated wrong peak
- (7) Other – Explain.

After review by the section manager, the manager will date and initial. Upon client request, the integrations will be reviewed by the QAU or his/her designee initialed and dated. The hard copies will be filed with the raw data.

**8 Calculations**

8.0 The sample and spikes are calculated against the initial calibration curve for the native PCDD/PCDF using isotope dilution. The C13 labeled surrogates are calculated using the two Internal Standards.

8.1 Quantitation algorithm check: The quantitation of the analytes is performed by the Waters Data System. The algorithm is checked at least once per computer file (daily) by calculating the amount of analyte injected from the peak response, using the calibration curve. The following calculation is used to check the quantitation:

$$\text{Concentration (ppt) Isotope Dilution Technique} = \frac{(A_s)(V_e) C_L \times D}{(A_i)(RF)(V_o)}$$

(Used to quantitate the 27 Toxic/LOC CBs)

$$\text{Concentration (ppt) Internal Standard Technique} = \frac{(A_s)(V_e) C_L \times D}{(A_a)(RF)(V_o)}$$

(Used to quantitate the remaining 182 CBs)

Where:

- A<sub>s</sub> = Sum Ion Abundance of analyte
- A<sub>i</sub> = Sum Ion Abundance of internal standard
- A<sub>a</sub> = Average Ion Abundance of internal standard
- C<sub>L</sub> = Labeled Congener Concentration (added to each sample)
- RF = Relative Response Factor of analyte from ICAL (see Sect 7.5.6)
- V<sub>o</sub> = Volume of sample extracted (L or Kg)
- V<sub>e</sub> = Final volume of extract (mL)
- D = Dilution factor if a dilution was made

8.2 Reporting Positive Findings: The Target Lynx software calculates the LOD and EMPC values, which are dynamic and dependent on the S/N ratio of each sample. The laboratory establishes the PQL, and it is a static value based on the lowest point in the calibration curve. J-values are reported between the LOC or EMPC and the PQL according to the following criteria:

8.2.1 If there is no peak present in the sample, then report "not detected" along with an LOD (or EDL) value in the EMPC/EDL column in LabWorks.



- 8.2.2 If there is a peak present in the sample, but it does not meet all the criteria for RRT or Isotope Ratio, then report "not detected" along with an EMPC value.
- 8.2.3 If there is a peak in the sample that is a confirmed hit above the PQL, then report that value with "NA" listed in the EMPC/EDL column in LabWorks.

8.2.4 LOD (Limit of Quantitation) or EDL (Estimated Detection Limit)

$$\text{LOD/EDL} = 2.5 \times Q_{is} \times (H_x^1 + H_x^2) \times D / V \times (H_{is}^1 + H_{is}^2) \times \text{RRF}$$

Where:

- $H_x^1$  and  $H_x^2$  = peak heights of both quantitation ions of the noise
- $H_{is}^1$  and  $H_{is}^2$  = peak heights of both quantitation ions of the appropriate IS
- D = Dilution Factor
- $Q_{is}$  = quantity of the appropriate IS injected (pg)
- $\text{RRF}_n$  = calculated RRF from the CCV
- V = volume or weight of sample extracted

8.2.5 EMPC (Estimated Maximum Possible Concentration)

$$\text{EMPC} = Q_{is} \times (A_x^1 + A_x^2) \times D / V \times (A_{is}^1 + A_{is}^2) \times \text{RRF}$$

Where:

- $A_x^1$  and  $A_x^2$  = areas of both quantitation ions of the noise
- $A_{is}^1$  and  $A_{is}^2$  = integrated areas of both quantitation ions of the appropriate IS
- D = Dilution Factor
- $Q_{is}$  = quantity of the appropriate IS injected (pg)
- $\text{RRF}_n$  = calculated RRF from the CCV
- V = volume or weight of sample extracted

8.2.6 PQL (Practical Quantitation Limit)

$$\text{PQL} = \text{CS}_1 \times E \times D$$

Where:

- $\text{CS}_1$  = concentration of low calibration standard (pg/mL)
- E = extraction ratio (for waters: 0.05mL / 1L) and (for soils (0.05mL / 10g)
- D = Dilution Factor

9 Quality Control

- 9.1 The laboratory must demonstrate initial proficiency study (IDP study) for solid and aqueous matrices, by generating data of acceptable accuracy and precision for target analytes using four replicate DI water spikes that have undergone the entire preparation, extraction and cleanup procedures outlined in the method and respective APPL SOPs. The laboratory must also repeat the following operations whenever new staff is trained or significant changes in instrumentation are made. See Table 1 of this SOP for recovery and %RSD limits for the IDP.
- 9.2 Sample quality control for preparation and analysis include the analysis of a method blank, a matrix spike/matrix spike duplicate, laboratory control sample, and sample



- duplicate in each analytical batch of 20 samples, and the addition of labeled internal standards and labeled C13 surrogates to each sample and QC sample.
- 9.3 The method blank must be shown to contain no analytes of interest above  $\frac{1}{2}$  the RL. If sample volume allows, samples with hits of analytes detected above  $\frac{1}{2}$  the RL in the method blank must be reanalyzed.
  - 9.4 The laboratory control sample will be included with each batch of 20 samples or less. The LCS consists of an aliquot of control matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate. See Table 1 in this SOP for compounds, spike levels and spike acceptance criteria. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
  - 9.5 The matrix spike and matrix spike duplicate will be included with each sample batch as per client. The client is to determine which sample is designated with MS/MSD. The %RPD limit for the MS/MSD is  $\leq 20\%$ . If the criteria is not met notify the project manager who will examine the DQOs and contact the client.
  - 9.6 Any native hits associated in the sample and sample duplicate, must have a %RPD limit of  $\leq 25\%$ . For DoD clients the acceptance limits for sample and sample duplicate must be  $\leq 20\%$ .
  - 9.7 If the recoveries of the LCS, MS/MSD, or sample duplicate are not within limits, the following are required:
    - 9.7.1 Confirm that there are no errors in calculations or surrogate solutions. Also, check instrument performance.
    - 9.7.2 Examine chromatograms for interfering peaks and for integrated areas.
    - 9.7.3 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
    - 9.7.4 Re-extract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration".
  - 9.8 The EPA 8290<sup>1</sup> method does not have an MDL requirement; however APPL Inc. will perform an initial MDL study for both soil and water extraction methods and cleanup procedures and then quarterly MDL checks. For DoD projects, the PQL will be increased to meet the PQL = 3 X MDL requirement, however the samples will not be screened for J-values between the MDL and the PQL, since the method requires the dynamic reporting of LOD/EDL or EMPC values generated by the quantitation software. These values are significantly lower than the PQL.
  - 9.9 Deviations: Any activity not performed in accordance with laboratory procedures or Quality Assurance Project Plans is considered a deviation from plan. All deviations from plan will be documented as to the extent of, and reason for, the deviation.
  - 9.10 Corrective Action: Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria will be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken is documented on a Quality Control Exception Report (QCER) and/or a Corrective Action Report (CAR).
  - 9.11 Data Reporting Criteria: Data is obtained from the primary column. If all the isomers are resolved within 25% using the DB-5 column, then the DB-225 confirmation column is not needed.



### 10 Data Validation

- 10.0 When QC parameters are exceeded, the following will take place: When the matrix spikes are outside of the limits they are re-digested and re-analyzed. When the LCS is outside of limits the entire batch is re-digested and re-analyzed. If there is not enough sample for re-digestion the Project Manager is notified whom in turn notifies the client by phone or fax. The case narrative or case letter explains the sequence of events and the data is qualified. If the calibration parameters are not met the standards are re-prepared and reanalyzed.
- 10.1 The analyst completing the work first reviews data. The initial calibration curve is reviewed, the continuing calibration %D is reviewed, the spike recovery and precision is reviewed, the performance check solution is reviewed, and the closing continuing calibration %D is reviewed. If at any point the review shows an out of control situation, the section manager is notified verbally and the problem is investigated. The correction may be one of several points considered: standard preparation, improper injection size, extraction technique, etc. The problem is potentially solved and reanalysis or re-extraction/reanalysis is completed.
- 10.2 The second level of review is either by a peer in the same section or the section manager. There is a Multilevel Quality Control Sign Off worksheet that is filled out in its entirety by the review person.
- 10.3 When QC parameters are exceeded, the following will take place: When the matrix spikes are outside of the limits they are re-injected and the client is notified. When the LCS is outside of limits the entire batch is re-extracted and reanalyzed. If there is not enough sample for re-extract, the Project Manager is notified who in turn notifies the client by phone, fax, or e-mail. The case narrative or case letter explains the sequence of events and the data is qualified. If the calibration parameters are not met, the standards are re-prepared and reanalyzed.
- 10.4 TargetLynx is the program used to quant all generated data. Consequently, flagging of data is mostly done using the TargetLynx program. This is flagged either with a "No" or "Yes". In TargetLynx, the question asked when checking tolerance such is the target ratio is "Does the measured value fall outside the specified tolerance?" or in essence, "Does the test fail?" Thus the value outside of the tolerance is flagged with a "Yes." Those within tolerance are flagged with a "No." The program does not allow changing of the flags from YES/NO to FAIL/PASS. This information is noted to avoid any confusion involved in flagging of data.

### 11 Pollution Prevention

All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents

### 12 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and lands disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.



### 13 Contingencies for Handling Out of Control or Unacceptable Data

In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a QCER. The QAU has the final authority to approve the actions taken

### 14 Deviations from the method

This SOP was compared to EPA method 8290<sup>1</sup>. If the TGDF isomers listed in the method are resolved by 25% on the DB-5 column, then the confirmation column analysis is not necessary for 2.3.7.8-TCDF. The column performance mix purchased by APPL Inc. contains 2.3.4.7-TCDF and 1.2.3.9-TCDF in order to show the separation needed to eliminate the confirmation column analysis.

### 15 Health and Safety (Sharon needs to add wipe tests and GC split vent to MeOH)

- 15.0 Lab coats and gloves are use at all times. All personnel handling raw samples must have been vaccinated or tittered for infectious disease. Follow all safety procedures as describes in the SOPs for samples suspected of containing biological hazards. Refer to APPL Inc. SOP SAFETY8290 for further procedures.
- 15.1 2.3.7.8-TCDD has been classified as a known human or mammalian carcinogen and teratogen. On the basis of the available toxicological and physical properties of the Dioxins and Furans, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks. Standards should be prepared using gloves, goggles and lab coats under a fume hood. Soil sample homogenization should be performed under a hood to reduce inhalation of Dioxin particles.
- 15.2 The pure standards and samples suspected to contain these compounds are handled using essentially the same techniques employed in handling radioactive or infectious materials. Well-ventilated, controlled access laboratories are required. Assistance in evaluating the health hazards of particular laboratory conditions may be obtained from certain consulting laboratories and from State Departments of Health or Labor, many of which have an industrial health service.



# Standard Operating Procedure

## QA Control Copy # 3

SOP: HPL8290  
Section: 10  
Revision: 4  
Date: 09/16/09

### SALUTATION

This procedure applies to all personnel who analyze extracts for Dioxins and Furans by EPA Method 8290<sup>1</sup>.

HPLC LABS  
CONFIDENTIAL

Section Manager: Sharon Oelshaw

Date: 9-18-09

QAU Director: Frances Sedaini

Date: 9/18/09

#### References:

<sup>1</sup>Method 8290, Revision 0, September 1994: Polychlorinated Dibenzodioxins (PCDDs) and Poly chlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography High Resolution Mass Spectrometry (HRGC/HRMS).

<sup>2</sup>CFR 40 Part 136, Appendix B, Chapter 1 (7-1-95 Edition), Revision 1.11



**TABLE 1**  
 Calibration Standard Concentrations and  
 Acceptance Criteria for CCV, LCS and IDP Study

ANALYTE	ICAL 0.1 ng/ml	ICAL 1 ng/ml	ICAL 2 ng/ml	ICAL 3 ng/ml	ICAL 4 ng/ml	ICAL 5 ng/ml	CCV  % D	IDP Study		LCS  % REC	Labeled C13 surrog % REC (In sample)
								% REC	% REC		
2,3,7,8-TCDD	0.1	1.0	2.5	10	50	200	20	70-130	70-130	*	
2,3,7,8-TCDF	0.1	1.0	2.5	10	50	200	20	70-130	70-130	*	
1,2,3,7,8-PeCDD	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,7,8-PeCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
2,3,4,7,8-PeCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,4,7,8-HxCDD	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,6,7,8-HxCDD	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,7,8,9-HxCDD	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,4,7,8-HxCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,6,7,8-HxCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,7,8,9-HxCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
2,3,4,6,7,8-HxCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,4,6,7,8-HpCDD	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,4,6,7,8-HpCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
1,2,3,4,7,8,9-HpCDF	0.25	2.5	6.25	25	125	500	20	70-130	70-130	*	
OCDD	0.5	5.0	12.5	50	250	1000	20	70-130	70-130	*	
OCDF	0.5	5.0	12.5	50	250	1000	20	70-130	70-130	*	

**Labeled Surrogate Standards**

C13-2,3,7,8-TCDD	50	50	50	50	50	50	30	40-135	*	40-135
C13-2,3,7,8-TCDF	50	50	50	50	50	50	30	40-135	*	40-135
C13-1,2,3,7,8-PeCDD	50	50	50	50	50	50	30	40-135	*	40-135
C13-1,2,3,7,8-PeCDF	50	50	50	50	50	50	30	40-135	*	40-135
C13-1,2,3,6,7,8-HxCDD	125	125	125	125	125	125	30	40-135	*	40-135
C13-1,2,3,6,7,8-HxCDF	125	125	125	125	125	125	30	40-135	*	40-135
C13-1,2,3,4,6,7,8-HpCDD	125	125	125	125	125	125	30	40-135	*	40-135
C13-1,2,3,4,6,7,8-HpCDF	125	125	125	125	125	125	30	40-135	*	40-135
C13-OCDD	250	250	250	250	250	250	30	40-135	*	40-135

**C13 Labeled Internal Standards**

C13-1,2,3,4-TCDD <sup>A</sup>	50	50	50	50	50	50	30	*	*	*
C13-1,2,3,7,8,9-HxCDD <sup>B</sup>	125	125	125	125	125	125	30	*	*	*

<sup>A</sup> Used to determine the recoveries of the TCDD, TCDF, PeCDD and PeCDF surrogates.

<sup>B</sup> Used to determine the recoveries of the HxCDD, HxCDF, HpCDD, HpCDF and OCDD surrogates



**TABLE 2**  
**GC Performance Check Standard**

Analyte	No. Cl Atoms	Concentration ng/ml
1.3.6.8-TCDD and -TCDF	4	10
1.2.8.9- TCDD and -TCDF	4	10
1.2.3.4 and 1.2.3.7-TCDD	4	10
1.2.3.8 and 1.2.3.9 and 2.7.3.7.8-TCDD	4	10
C13-2.3.7.8 -TCDD	4	50
1.2.4.6.8 and 1.2.4.7.8-PeCDD	5	25
1.3.4.6.8-PeCDF	5	25
1.2.3.8.9-PeCDD and -PeCDF	5	25
1.2.4.6.7.9 and 1.2.4.6.8.9-HxCDD	6	25
1.2.3.4.6.7-HxCDD	6	25
1.2.3.4.6.8 and 1.2.3.4.8.9-HxCDF	6	25
1.2.3.4.6.7.9-HpCDD	7	25
1.2.3.4.6.7.8-HpCDD and -HpCDF	7	25
1.2.3.4.7.8.9-HpCDF	7	25
1.2.3.4.6.7.8.9-OCDD and -OCDF	8	50

**TABLE 3**  
**C13 Internal Standards and Recovery Standards**

Analyte	Internal Standard Concentration ng/ml	Recovery Standard Concentration ng/ml
C13 -2.3.7.8.-TCDD	10	----
C13 -2.3.7.8.-TCDF	10	----
C13 -1.2.3.7.8.-PeCDD	10	----
C13 -1.2.3.7.8.-PeCDF	10	----
C13 -1.2.3.6.7.8.-HxCDD	25	----
C13 -1.2.3.6.7.8.-HxCDF	25	----
C13 -1.2.3.4.6.7.8.-HpCDD	25	----
C13 -1.2.3.4.6.7.8.-HpCDF	25	----
C13 -OCDD	50	----
C13 -1.2.3.4-TCDD	----	50
C13 -1.2.3.7.8.9-HxCDD	----	50



**TABLE 4**  
**Dioxin and Furan Spike Mix**

Analyte	Concentration ng/ml
2,3,7,8-TCDD	100
2,3,7,8-TCDF	100
1,2,3,7,8-PeCDD	250
1,2,3,7,8-PeCDF	250
2,3,4,7,8-PeCDF	250
1,2,3,4,7,8-HxCDD	250
1,2,3,6,7,8-HxCDD	250
1,2,3,7,8,9-HxCDD	250
1,2,3,4,7,8-HxCDF	250
1,2,3,6,7,8-HxCDF	250
1,2,3,7,8,9-HxCDF	250
2,3,4,6,7,8-HxCDF	250
1,2,3,4,6,7,8-HpCDD	250
1,2,3,4,6,7,8-HpCDF	250
1,2,3,4,7,8,9-HpCDF	250
OCDD	500
OCDF	500

**TABLE 5**  
**Theoretical Ion Abundance Ratios and QC Limits**

Cl Atom	m/z Forming Ratio	Theoretical Ratio	Ratio Acceptance Range
4	$m/m+2$	0.77	0.65 - 0.89
5	$m/(m+2)$	1.55	1.32 - 1.78
6	$(m+2)/(m+4)$	1.24	1.05 - 1.43
6	$m/(m+2)$	0.51	0.43 - 0.51
7	$(m+2)/(m+4)$	1.04	0.88 - 1.20
7	$m/(m+2)$	0.44	0.37 - 0.51
8	$(m+2)/(m+4)$	0.89	0.76 - 1.02



TABLE 6  
2.3.7.8-TCDD Toxicity Equivalency Factors (TEFs)<sup>1</sup>

Analyte	TEF
2.3.7.8-TCDD	1.00
1.2.3.7.8-PeCDD	0.50
1.2.3.6.7.8-HxCDD	0.10
1.2.3.7.8.9-HxCDD	0.10
1.2.3.4.7.8-HxCDD	0.10
1.2.3.4.6.7.8-HpCDD	0.01
1.2.3.4.6.7.8.9-OCDD	0.001
2.3.7.8-TCDF	0.1
1.2.3.7.8-PeCDF	0.05
2.3.4.7.8-PeCDF	0.5
1.2.3.6.7.8-HxCDF	0.1
1.2.3.7.8.9-HxCDF	0.1
1.2.3.4.7.8-HxCDF	0.1
2.3.4.6.7.8-HxCDF	0.1
1.2.3.4.6.7.8-HpCDF	0.01
1.2.3.4.7.8.9-HpCDF	0.01
1.2.3.4.6.7.8.9-OCDF	0.001



**Appendix A**

**Definitions**

**Calibration standard** - A solution prepared from the primary dilution standard solution or stock standard solution and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration.

**Extracted Ion Current Profile (EICP)**—The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundance versus time or scan number.

**Field Reagent Blank** - 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, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

**Instrument blank (BIK)** - An aliquot of reagent water or other blank matrix to demonstrate that the instrument is not contributing contaminants to the samples.

**Internal Standard (IS)** - 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.

**Instrument Performance Check (IPC)** - A solution of one or more compounds (analytes, surrogate, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

**Laboratory control spike (LCS)** - An aliquot of reagent water or other matrix to which known quantities of the method analytes are added in the laboratory. The LCS 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.



**Appendix A con't**

**Laboratory Reagent 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 LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

**Limit of Detection** - 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. 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%)

**Limit of Quantitation** - The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence. 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. This also equates with the term Practical Quantitation Limit (PQL).

**Matrix** - A surrounding substance within which something originates, develops, or is contained, such as: drinking water, saline/estuarine water, aqueous substance other than drinking water or saline/estuarine water, non-aqueous liquid, biological tissue, solids, soils, chemical waste, and air.

**Matrix duplicate (MD)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix sample and matrix sample duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**Matrix spike (MS)** - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The matrix spike 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 matrix spike corrected for background concentrations.



**Appendix A con't**

**Matrix spike duplicate (MSD)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a matrix spike and matrix spike duplicate, indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

**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, the reagents, or the apparatus.

**Method detection limit** - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, as determined from analysis of a sample containing the analyte in a given matrix, as described in 40 CFR Part 136, Appendix B, 1 July 1995 edition.

**Practical quantitation limit** - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The practical quantitation limit is generally three to ten times greater than the method detection limit.

**Primary Dilution Standard** - A solution of several analytes prepared in the laboratory from stock solution and diluted as needed to prepare calibrations solutions and other needed analyte solutions.

**Quality Control Sample (QCS)** - A solution of method analytes of known concentrations which is used to fortify an aliquot of LCS 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.

**Sample Duplicate (DUP1/DUP2)** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analytes of DUP1/DUP2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures



Appendix A con't

**Stock Standard Solution** - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials purchased from a reputable commercial source.

**Surrogate** - 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 surrogate is to monitor method performance with each sample.

APPLIED  
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# Standard Operating Procedure

## PCDD's and PCDF's (EPA METHOD 8290<sup>1</sup>) SEPARATORY EXTRACTION of Aqueous Samples

### STATEMENT OF PURPOSE

This procedure describes the separatory funnel extraction for aqueous samples for Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) analysis by high-resolution GC/MS. The extraction code for this method is SEP8290.

### INSTRUCTIONS

#### 1.0 Scope and Application

- 1.1. This SOP describes a procedure for isolation and concentration of PCDD and PCDF in aqueous matrices. This procedure also describes the concentration techniques suitable for preparing the extract for the EPA 8290<sup>1</sup> cleanup or for direct analysis by HR GC/MS if cleanup is not required.
- 1.2. 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.
- 1.3. If an individual project has its own QAPP with client specific requirements that are different than the SOP, the QAPP overrides the SOP. This information will be specified in the comment section of the ARF.

#### 2.0 Method Summary

- 2.1. A measured amount of sample, usually 1 liter of aqueous sample, is extracted with the appropriate solvent using separatory funnel extraction.
- 2.2. The extract is macro-concentrated by Rotovap and, as necessary, exchanged into a solvent or column compatible with the cleanup to be used.
- 2.3. The eluent from the final cleanup is micro-concentrated under nitrogen and brought to a final volume of 50 $\mu$ L.

#### 3.0 Sample Preservation, Containers, Handling and Storage

- 3.1. Aqueous samples should be collected in 1 liter glass jars and stored at 4°C  $\pm$  2°C until delivery to the laboratory.
- 3.2. When the samples are delivered to the laboratory they are placed into a refrigerator in the sample receiving area that is kept at 4°C  $\pm$  2°C.
- 3.3. Samples must be extracted within 30 days of sampling.
- 3.4. Extracts must be stored under refrigeration in the dark. Extracts must be analyzed within 45 days of extraction.

#### 4.0 Interferences and Potential Problems

- 4.1. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under conditions of the analysis by analyzing method blanks. All solvents used are lot tested for acceptability prior to use.
- 4.2. A 4L container of each solvent lot is received by the laboratory. The lot number is prepared per SOP ORG041 and the extract is given to the appropriate section for the



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determinative step. If the results of the extract have no target analytes above the MDL, the lot number is accepted.

- 4.3. Phthalates esters contaminate many types of products commonly found in the laboratory. Serious phthalate contamination can occur if consistent quality control is not practiced.

### 5.0 Equipment/Apparatus

- 5.1. Nitrogen blowdown apparatus
- 5.2. Balances capable to accurately weighing to 0.01g and 0.0001g
- 5.3. Centrifuge
- 5.4. Water bath with temperature controlled within  $\pm 2^{\circ}\text{C}$
- 5.5. Glove box
- 5.6. Stainless steel spoons and spatulas
- 5.7. Laboratory hoods
- 5.8. Pipettes, disposable, Pasteur, 150mm long X 5mm ID
- 5.9. Pipettes, disposable, serological, 25mL
- 5.10. Glass injection vials (0.3mL conical)
- 5.11. Mortar and Pestle
- 5.12. Blender
- 5.13. 6mL SPE glass tubes
- 5.14. Glass fiber filters (Whatman 0.70 $\mu\text{m}$ )
- 5.15. Glass funnels
- 5.16. Desiccator
- 5.17. Rotary evaporator and water bath (with micro-concentration adaptor when needed)
- 5.18. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar
- 5.19. 500mL flat bottomed boiling flasks
- 5.20. Silica beads (100 mesh)
- 5.21. Assorted Glass "A" syringes
- 5.22. SPE Vacuum Manifold Apparatus
- 5.23. Soxhlet extractor apparatus
- 5.24. Soxhlet thimbles
- 5.25. Conical glass centrifuge tube (15mL)
- 5.26. Erlenmeyer Flask (250mL, 1000mL)
- 5.27. Explosion proof hot plate
- 5.28. Aluminum foil
- 5.29. Laboratory oven (capable of sustaining temperatures up to 400 $^{\circ}\text{C}$ )
- 5.30. 125mL separatory funnel (for soil back-extraction)
- 5.31. Supelco Custom SPE Cartridges (Carbon/Celite)
- 5.32. Supelco Custom SPE Cartridges (Acid/Base Silica Gel)

### 6.0 Reagents.

- 6.1. Organic free water
- 6.2. Quartz sand (or Ottawa Sand)
- 6.3. Concentrated Sulfuric acid – reagent grade
- 6.4. 20% Potassium Hydroxide solution - (20g KOH dissolved into 100mL DI Water)
- 6.5. 5% Sodium Chloride solution – (5g NaCl dissolved into 100mL of DI Water)
- 6.6. Methylene chloride – reagent grade
- 6.7. Nonane – reagent grade



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- 6.8. Toluene – reagent grade
- 6.9. Hexane – reagent grade
- 6.10. Acetone – reagent grade
- 6.11. Methanol – reagent grade
- 6.12. Cyclohexane – reagent grade
- 6.13. Activated Silica Gel-100 mesh (methylene chloride rinsed and baked @ 180°C for 1 hour: cooled in Desiccator and stored in glass jar with Teflon-screw cap)
- 6.14. Acidic Silica Gel (100g activated silica gel + 44g concentrated H<sub>2</sub>SO<sub>4</sub> mixed well and stored in glass jar with Teflon-screw cap)
- 6.15. Basic Silica Gel (100g activated silica gel + 30g of 1N NaOH mixed well and stored in glass jar with Teflon screw cap)
- 6.16. Celite 545 (stored in a sealed container at room temperature)
- 6.17. Active carbon AX-21 (prewashed with methanol and dried in vacuum at 110°C. Store in a glass bottle sealed with a Teflon lined screw cap.
- 6.18. Activated Anhydrous Sodium Sulfate (methylene chloride rinsed and baked @ 400°C for 1 hour: cooled in Desiccator and stored in glass jar with Teflon-screw cap)

### 7.0 Procedure:

#### 7.1. Preparation for Extraction:

##### 7.1.1 Preparation of Aqueous Samples:

- 7.1.1.1 Allow time for sample to reach room temperature.
- 7.1.1.2 Mark the water meniscus of the sample bottle to later determine the exact sample volume.
- 7.1.1.3 Spike the samples, blanks, LCS and MS/MSD with the acetone diluted surrogate solution. Spike the LCS and MS/MSD with spike mix.

**NOTE:** The addition of spike and surrogate will be witnessed by a second person and will be documented on the extraction sheet.

- 7.1.1.4 For samples with >1% solids, filter through a 0.45µm glass fiber filter that has been rinsed with toluene. If there is too much solids to filter, then centrifuge sample, decant and then filter the aqueous phase.
- 7.1.1.5 Combine the solids from the filter paper and the centrifuge contents and proceed to the extraction process for soils/sediment.
- 7.1.1.6 Pour the aqueous filtrate into a 2L separatory funnel. Rinse out the sample bottle with 60mL methylene chloride and add the liquid to the separatory funnel. Shake the funnel for two minutes with periodic venting.
- 7.1.1.7 Allow the organic layer to separate from the water phase. Making sure the emulsion interface between layers is not more than one third the volume of the solvent layer.
- 7.1.1.8 Pass the methylene chloride through a filter funnel packed with a glass wool plug and 5g anhydrous sodium sulfate into a Rotavap flask.
- 7.1.1.9 Repeat the extraction twice with fresh 60mL portions of methylene chloride. After the third extraction, rinse the sodium sulfate with an additional 30mL methylene chloride to ensure quantitative transfer.
- 7.1.1.10 Concentrate the extract in a rotary evaporator (35°C water bath), to a volume of 5mL. Allow it to cool.
- 7.1.1.11 Add 50mL hexane and (if applicable) the concentrate obtained from Section 7.1.1.5 and concentrate down to 5mL..



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- 7.1.1.12 Transfer the concentrate to a 125mL separatory funnel.
- 7.1.1.13 Rinse the flask and the lower joint with two 5mL portions of hexane and combine the rinses with the extract to ensure quantitative transfer.
- 7.1.1.14 Determine the original sample volume of the sample bottle using a 1000mL graduated cylinder. Record the sample volume to the nearest 5mL.
- 7.1.1.15 Proceed to cleanup stage.

## 7.2. Cleanup Stage

### 7.2.1. Acid/Base Partition

- 7.2.1.1. Add 50mL of the H<sub>2</sub>SO<sub>4</sub> solution from sect 6.3. Shake for 2min and discard the aqueous layer. Repeat the "acid washing" until no color is visible in the aqueous layer for a maximum of 4 washings.
- 7.2.1.2. Add 40mL of NaCl solution from sect 6.5. Shake for 2min and discard the aqueous layer.
- 7.2.1.3. Add 40mL of 20% potassium hydroxide from sect 6.4. Shake for 2min and discard the aqueous layer. Repeat the "base washing" until no color is visible in the aqueous layer for a maximum of 4 washings.
- 7.2.1.4. Add 40mL of NaCl solution from sect 6.5. Shake for 2min and discard the aqueous layer.
- 7.2.1.5. Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a Rotovap flask. Rinse the funnel with the sodium sulfate with two 15mL portions of hexane, add the rinses to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (if any) are removed.

### 7.2.2. Silica Column Cleanup (Supelco Custom Silica Gel SPE Cartridges may be used for this step, or the technician may prepare his own column as follows):

- 7.2.2.1. Preparation of Silica/Alumina Columns for sample cleanup (as needed). Pack SPE glass tube listed in sect 5.13 with the following materials from bottom to top:

- Glass wool plug
- Activated silica gel (1.0g) – see sect 6.15
- Basic silica gel (2.0 g) – see sect 6.17
- Acidic silica gel (4.0g) – see sect 6.16
- Activated silica gel (2.0g) – see sect 6.15

- 7.3.2.1 Elute the column with 10mL hexane. Be careful not to let the column run dry.
- 7.3.2.2 Dissolve the residue from 7.3.1.5 in 2mL of hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4mL) to complete transfer of sample. Elute the silica gel column with 90mL of hexane. Concentrate the eluent on a rotary evaporator (35°C water bath) to approximately 1mL.

### 7.3.3 Carbon Column Cleanup (Supelco Custom Carbon SPE Cartridges may be used for this step, or the technician may prepare his own column as follows):

- 7.3.3.1 Preparation of an AX-21/Celite 545 column for sample cleanup (as needed): Mix 5.4g active carbon Ax-21 (Sect 6.19) and 62.0g Celite 545 (Sect 6.18). Activate the mixture at 130°C for 6 hours and store it in a desiccator. Using a triangular file, score the ends of a 25mL



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disposable serological pipette. Carefully snap off the ends and pack with the following materials from bottom to top:

- Glass wool plug
- 1cm plug of Celite 545 (see Sect 6.18)
- 1cm plug of the AX-21/Celite 545 Mixture (see Sect 7.2.3.1)
- 1cm plug of Celite 545 (see Sect 6.18)
- Glass wool plug

Note: The following elution procedure may be used with laboratory-made carbon columns or the purchased SPE carbon cartridges, without the use of a SPE vacuum manifold.

- 7.3.3.2 Rinse the AX-21/Celite 545 column with 5mL of toluene.
- 7.3.3.3 Rinse with 2mL of (75:20:5, v/v) methylene chloride/methanol/toluene solution.
- 7.3.3.4 Rinse with 1mL of (1:1, v/v) Cyclohexane/methylene chloride solution.
- 7.3.3.5 Rinse with 5mL of hexane.
- 7.3.3.6 The flow rate should be less than 0.5mL/min. Discard the rinses.
- 7.3.3.7 While column is still wet with hexane, add the sample concentrate.
- 7.3.3.8 Rinse the concentrator tube twice with 1mL hexane.
- 7.3.3.9 Rinse column sequentially with two 2mL portions of hexane, 2mL Cyclohexane/methylene chloride (50:50, v/v), and 2mL-methylene chloride/methanol/toluene (75:20:5, v/v). Combine these eluents: This combined fraction may be used as a check on column efficiency.
- 7.3.3.10 Turn the column upside down and elute the PCDD/PCDF fraction with 20mL of toluene. Add the rinse to the eluent.
- 7.3.3.11 Concentrate the extract in a rotary evaporator (50°C water bath), to near dryness. Add 1mL of hexane.
- 7.3.4 **Micro-Concentration:** Quantitatively transfer the sample extract from the boiling flask to a 15mL centrifuge tube. Rinse out flask with two 5mL portions of hexane and add it to the centrifuge tube. Assemble the extracts in a nitrogen blow-down apparatus.
  - 7.3.4.1 Adjust the flow of the Nitrogen gas until the surface of the solvent is just visibly disturbed. When the volume of the eluent has concentrated to approximately 100 $\mu$ L, transfer the concentrate extract into a 300 $\mu$ L conical injection vial (marked at the 50 $\mu$ L level) for further concentration. Rinse centrifuge tube three times with 300 $\mu$ L of hexane. Between rinses, continue concentrating to 100 $\mu$ L volume and transfer concentrate to injection vial.
  - 7.3.4.2 Add 30 $\mu$ L of nonane and 20 $\mu$ L Internal Standard in nonane to the extract and continue concentrating under nitrogen until the 50 $\mu$ L level is reached. The sample is now ready for instrument analysis (see SOP ANA8290).

## 8.0 Calculations - NA

## 9.0 Quality Control

- 9.1 Demonstration of Capability (DOC) – You must demonstrate initial proficiency by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This consists of preparing four lab controls with a second source standard, or spike prepared independently of the calibration. The DOC must also be performed for each of the cleanup steps listed in section 7.4.



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- 9.2 To all samples, blanks, laboratory control spikes (LCS) and matrix spikes (MS/MSD) add the surrogate solution and spiking solution as listed on the extraction sheet.
- 9.3 The Organic Extraction supervisor, the Section Manager, or a properly trained analyst will perform surrogate/spike additions.
- 9.4 Each batch of no more than 20 samples should include a Laboratory Control Sample (LCS) and a Laboratory Method Blank. These should consist of DI water and be extracted just like the samples.
- 9.5 Each batch should also include either a Matrix spike and an unspiked sample duplicate or a matrix spike and a matrix spike duplicate (MS/MSD) if the client has provided enough sample volume.

### 10.0 Data Validation - NA

### 11.0 Pollution Prevention

All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents.

### 12.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

### 13.0 Contingencies for Handling Out of Control or Unacceptable Data

In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a QCER. The QAU has the final authority to approve the actions taken.

### 14.0 Deviations to the method

This SOP was compared to method 8290. There are no deviations to the method.

### 15.0 Health and Safety

Lab coats safety glasses and gloves are used at all times. A face shield will be worn when handling glass separatory funnels. Some samples require the use of respirators. This is on a case by case basis.



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**SALUTATION**

This procedure is applicable to all personnel who perform separatory funnel extractions on water samples for EPA Method 8290<sup>1</sup> analysis.

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED

Section Manager: *[Signature]*

Date: 10-1-09

QAU Director: *[Signature]*

Date: 10/1/09

<sup>1</sup> USEPA Method 8290, Revision 0, September 1994. Polychlorinated Dibenzodioxins and Polychlorinated Dibenzodioxins by HRGMS



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Table 1

Dioxin/Furan Aqueous Extraction Flow Chart

AQUEOUS SAMPLES

SOP Sect

Add Spike and Surrogate	7.1.1.3
Filter samples with >1% solids	7.1.1.4
Separatory funnel extraction	7.1.1.6
Rotovap to 5.0 mL volume	7.1.1.10
Add 50mL hexane and Rotovap to 5mL	7.1.1.11
Acid Partition	7.2.1.1
NaCl Partition	7.2.1.2
Base Partition	7.2.1.3
NaCl Partition	7.2.1.4
Rotovap to near dryness	7.2.1.5
Silica Gel Cleanup	7.2.2.3
Rotovap to 1mL	7.2.2.3
Conc. under N <sub>2</sub> to 2mL	7.2.2.4
Carbon Cleanup	7.2.3.7
Rotovap to 1mL	7.2.3.11
Conc. under N <sub>2</sub> (20 $\mu$ L I.S. in Nonane)	7.2.3.12



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**Table 2**

**"Spike" and "Surrogate" solutions**

**Dioxin/Furan "Spike"**

20 $\mu$ L of CIL EDF-5008 spiked to a final volume of 20mL with ACETONE solvent

(Add 1.0mL of the Dioxin/Furan Spike Mix to each 1.0 Liter aliquot of LCS, MS/MSD)

**Dioxin/Furan "Surrogate"**

25 $\mu$ L CIL EDF-5005 spiked into each 1.0 Liter aliquot of Blank, LCS, MS/MSD, and SAMPLE

(Add 25 $\mu$ L of the Dioxin/Furan Surrogate to each 1.0 Liter aliquot of LCS, MS/MSD)

**Dioxin/Furan "Internal Standard"**

20 $\mu$ L of CIL EDF-4055 in Nonane solvent

(Add 20 $\mu$ L of the I.S. to each 100 $\mu$ L extract prior to micro-concentration of the Blank, LCS, MS/MSD and SAMPLE)

*Note 1: See APPL SOP ANA8290 for a list of the Dioxin/Furans present in each mix shown above.*



# Standard Operating Procedure

## RECEIVING SAMPLES

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### **STATEMENT OF PURPOSE**

This procedure describes the proper way for the sample custodian or his/her designee to receive samples at APPL, Inc. This SOP also describes shuttle temperature determination, sample acceptance policy and completion of the cooler receipt form, checking incoming samples for the presence of ionizing radiation, amending an Analysis Request Form (ARF), completing a sample receiving quality control check-off sheet, photocopying and distributing and ARF and re-receiving samples. This procedure also addresses the steps to take when analyses are cancelled.

### **INSTRUCTIONS**

The sample(s) are delivered to the personnel in the laboratory responsible for receiving samples, the sample custodian or his/her designee, referred to hereafter as "receiving".

Upon receipt, receiving will validate that the chain of custody has appropriate signatures from the shipper and courier(s). Receiving may relinquish shuttles for the courier when courier identity can be validated. The COC is signed at the first available space for a lab signature, dated and the time entered by the receiving personnel.

Samples that do not meet sample acceptance criteria are not received into the laboratory. (See Appendix B) The project manager is notified immediately who then notifies the client. All pertinent information regarding the rejection is conveyed to the client. The client is asked to remove the samples from the laboratory.

#### **1.0 Logging ice chests into the ice chest database**

- 1.1 Select the ARF Summary icon on the desktop of the computer.
- 1.2 Select receiving menu, and then select Ice Chest Check In/Out. Select check in.
- 1.3 Using the scanner, scan the ice chest bar codes located on the either side of the ice chest. If the number does not transfer to the cell, then look into the Ice Chest Menu on the previous Receiving window and look for the ice chest number to see if is on the list. If it is not, then it was not initially scanned as it went out of the building.
- 1.4 Using the scanner, scan the Fed Ex tracking number located on the top of the lid. The ice chest number should automatically show up on the window. If there is no Fed Ex numbers then type 999 in the cell.
- 1.5 When all the cells are filled in click on Save.

#### **2.0 Screening shuttle and contents for radiation**

- 2.1 Prior to the acceptance of samples by APPL, Inc., the sample shuttle will be screened for the presence of ionizing radiation. Using a "Ludlum Model 2 Survey Meter" and "Model 44-9 Wand", the sample custodian will screen the shuttle before opening the shuttle. To do this, turn the black knob to X0.1, take the plastic cover off of the scanning wand, and pass the wand around the shuttle. The background for this instrument is between 50-100 counts per minute. As a general rule, two times the background may be used as a cutoff for acceptance into the laboratory. Anything above that level, the sample custodian will contact the project manager for instructions.
- 2.2 If the shuttle passes this screening, it will be opened. Shuttles are to be opened under a hood. The paperwork is removed and the monitor will be used once



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again on the packing material and the unopened samples. If the container passes this screening, the sample custodian will temp the shuttle. If at any point in the screening process the container and/or samples fail the screening, the client will be immediately notified regarding disposition of the samples. (See SOP #SHR022)

- 2.3 The scanner and wand must be calibrated once per year. To do this, call Ludlum Measurements, Inc., 501 Oak Street, Sweetwater, TX. 79556 @ 1-915-235-5494 and obtain a Return Authorization Number. Send out on a Friday with same day service. You will need to use a PO number. Do this by using a PO #60.

### 3.0 Scanning the COC

- 3.1 Upon passing the RAD screen, shuttle temperatures for all appropriate projects, are measured and recorded on the Cooler Receipt Form.
- 3.2 When samples are not received immediately upon delivery to the laboratory (as may be the case when multiple sets arrives at the same time), the shuttles are opened and the chain of custody is removed. Samples with analyses with short holding times or samples that have little holding times remaining are given first priority, then rush samples. In this case, the receiving custodian will notify the correct section(s) or department(s) manager on the rushes or short hold time. Both the Section Manager and analyst will sign and date the Cooler Receipt Form. Check the Holding Times Sheet posted in Receiving to see a list of the hold time for the different analyses.
- 3.3 Assign an ARF number and scan the COC into the database. Write the ARF number on the ziplock bag containing the COC or directly on the upper left-hand corner of the COC.

### 4.0 Taking shuttle temperature

- 4.1 Wear gloves whenever handling samples. Wear safety glasses when opening shuttles.
- 4.2 Remove custody seals, if any are provided, and attach them to the Cooler Receipt form. (See Appendix A) Note: If one of the shuttles out of the group does not have a custody seal make a note of which shuttle and what sample bottles are affected on the Cooler Receipt form.
- 4.3 Upon receipt of a shuttle, open the lid and remove the Chain of Custody. Check the Chain of Custody to see if the QC level was noted or that the sampler listed a temperature blank on the Chain of Custody. If the client has provided a temperature blank the thermometer will be placed directly into the bottle to record the temperature.
- 4.4 If a temperature blank has not been provided a calibrated thermometer (NIST traceable certification) is placed in the ice chest among the sample containers. Make sure the thermometer does not come in direct contact with the wet or blue ice, if possible. If you are unable to immerse the thermometer in water then note on the cooler receipt form that you were unable to take the temperature due to lack of a temperature blank or cooler water and inform the Project Manager.
- 4.5 Close the lid and allow the contents to reach thermal equilibrium, (app. 5 minutes).
- 4.6 Reopen the lid and immediately record the temperature (in 0.5°C increments) on the Cooler Receipt form, after you have added or subtracted the correction factor



of the thermometer. Record the temperatures of each shuttle sent per project. (E.g. If the client sent 3 shuttles for project #XXX then there should be three temperatures noted.)

- 4.7 If the temperature exceeds the Maximum Allowable Temperature ( $\leq 6^{\circ}\text{C}$ ) notify the Project Manager immediately and record your conversation on the Cooler Receipt form. Make a note of what cooler and what sample bottles are affected on the Cooler Receipt form and on the COC.

### 5.0 Removing broken samples

- 5.1 Sample container integrity is checked and the sample container identities are compared to the chain of custody. The following steps should be taken if there are samples with broken containers in the shuttle.
- 5.2 Remove all of the unbroken sample containers from the shuttle.
- 5.3 Notify the Project Manager to determine if the contents are known to contain contaminated samples.
- 5.4 If it has been determined that the shuttle has not come from a waste treatment facility or hazardous waste site it can be removed to the back of the building by the dumpster. A picture of the broken sample(s) will be taken before disposal. Spill the contents onto ground to let the water and ice separate from the broken glassware.
- 5.5 Sweep the glass up with a dustpan and broom and place it in the Dumpster.
- 5.6 Rinse out the shuttle with a bucket of water and let dry outside.
- 5.7 If the shuttle is known to contain samples from a hazardous waste site as per the Project Manager, the shuttle is to be put aside until further steps are determined as to when the client will pick-up the shuttle.

### 6.0 Shuttle with missing and extra samples

- 6.1 If samples are missing, Receiving will notify the Project Manager and /or the Lab Director immediately and will have them search for the sample containers. The packing materials and ice will not be placed in the Dumpster until the Project Manager and/or Lab Director have signed the cooler receipt form.
- 6.2 If extra samples are received, Receiving is to notify the Project Manager and/or Lab Director. The extra samples are to be logged onto the Cooler Receipt Form and signed off by the Project Manager and/or the Lab Director.

### 7 Data acceptability

- 7.1 The following outlines the circumstances under which samples will be accepted. Data generated from any samples, which do not meet the following criteria, will be flagged. The nature and substance of the variation will be clearly defined in the case narrative. The following documentation will be noted on the chain of custody: If any of the following is not noted on the chain of custody, the Project Manager must be notified.
- 7.2 Sample Identification
  - the location, date and time of collection
  - collector's name
  - preservation added
  - matrix type
  - any special remarks concerning the sample



- 7.3 The samples will be identified with a label that is durable (water-resistant) and any markings made on the label will be with indelible ink.
- 7.4 Appropriate sample containers for the analyses requested will be used.
- 7.5 Adherence to specified holding times.
- 7.6 Adequate sample volume must be provided to perform the analyses requested.
- 7.7 Procedures to use when samples show signs of damage or contamination.
- 7.8 Proper preservation for the analyses requested.
- 7.9 If a Chain of Custody is not received or is incomplete to the point that analysis cannot proceed without further instruction, the project manager is notified. The project manager or sample receiving will notify the client by phone, e-mail or by fax, stating that the Chain of Custody was not received or is incomplete. The samples are then placed in the "A" refrigerator or if they are volatile they are placed in VOA refrigerator, until the client is able to respond to the information. Once the client has responded, the samples are then logged into Labworks and the rest of the process is completed.
- 7.10 When breakage occurs and causes insufficient volume for the analyses requested, the breakage is noted on the cooler receipt form, and the project manager is notified. If the project manager is unavailable the Lab Director is notified and the chain of custody is faxed to the client or the client is notified by phone.

**NOTE: ALL DISCREPANCIES SHOULD BE RESOLVED BEFORE THE SAMPLES ARE ASSIGNED FOR ANALYSIS. THIS INCLUDES SAMPLE IDS, SAMPLE DATE, HOLDING TIMES, TEMPERATURE, PRESERVATION, AIR BUBBLES LARGER THAN A PEA (VOA), INAPPROPRIATE SAMPLE CONTAINERS, INSUFFICIENT SAMPLE VOLUME, LEAKAGE, AND BROKEN OR CRACKED SAMPLE CONTAINERS. ALL DISCREPANCIES ARE DISCUSSED WITH THE PROJECT MANAGER WHO WILL CALL THE CLIENT FOR FURTHER INSTRUCTION. ALL DISCREPANCIES ARE NOTED ON THE ARF AND THE COOLER RECEIPT FORM. If one of the VOA containers comes in with an air bubble, then do not label that one with W01. Instead start with the one with no bubble then from the smallest to largest bubble.**

- 7.11 The samples are placed on the counter in rows according to the COC supplied by the customer. If the COC does not have the time of sampling written down and the bottles do, then transfer the time to the COC. An inventory of the containers received is documented on the APPL Sample Inventory Sheet. (Appendix C) This form categorizes the containers by type, size and preservation used by listing the number of containers received for each category. When samples for volatile analysis are received, the samples will only be at room temperature 5-10 minutes. If there is a time delay in receiving samples, the samples must be placed in a 4°C environment until the log-in process can be completed.
- 7.12 All client container labels will be checked against the COC. The client sample ID should be exactly the same as listed on the COC. If it does not match, the Lab Director or Project Manager will be notified.



7.13 After the APPL ID is placed on the container the Sample Custodian will double-check the APPL ID against the client ID, then will put his/her initials on the ID for W01, S01 or M01, before it is placed into the refrigerator.

**8 Completing the cooler receipt form**

8.1 The cooler receipt form (Appendix A) is completed for every group of samples received. This form documents the condition of the cooler(s) and the samples upon arrival to the laboratory.

8.2 The project manager will be informed of any abnormalities or departures from the acceptance policy. The sample receiving personnel or the project manager will inform the client of deviations from the relevant test method. Any communication with the client will be documented on the form. The deviations will be documented in the case narrative.

**9 Checking the pH of samples**

9.1 The samples are removed from the cooler. The pH is checked on all preserved aqueous samples (preserved plastic bottles). VOA vials are checked after completion of analysis.

9.2 The container is inverted several times to mix the sample.

9.3 To check the pH when the sample is acidified below pH 4 use Baker pH indicator strips, which are in the range of 1-7-3-8.

9.4 To check the pH for basic samples above pH 3.8 use EMD pH indicator strips with a range of 0-14.

9.5 The sample container lid is removed, a small amount of the sample is poured into a disposable cup and a pH strip is used to test the sample from the cup. The pH strip is **NOT** inserted into the sample container. The tested sample is **NOT** returned to the sample bottle.

9.6 The pH of the sample is noted on the top or side of the sample bottle.

**10 Generating the Analysis Request Form (ARF)**

10.1 Any sample that is placed on hold or does not have an any analysis requested is entered at the end of the set. Enter sample ID in the order it is listed on C.O.C.

10.2 Samples with quick turn are placed on a separate ARF and are logged in first.

10.3 Open Labworks. Enter your username and password.

10.4 Select RECVI.MLT

10.5 Select Multi-Sample Login

10.6 Fill in the following information:

10.6.1 On the first cell under Client code, right click. Choose Select Client Name. Type in the first few letters of the client's name and highlight the name you have selected. The Client's name, address, etc. will show up on line one of the Labworks Multi-login page. Check the address, etc. against the COC.

10.6.2 Report Format – Usually this has a default setting for each client. Make sure it matches what is on the COC. If there is none listed then type in Std.

10.6.3 Matrix - Water, Soil, or Misc. These words must be typed in exactly as they are written here or they will not be accepted by Labworks.



- 10.6.4 Delivered - Enter the name of the courier delivering the samples.
- 10.6.5 Sample Date - This should be on the COC or on the bottles.
- 10.6.6 Collection Time - This should be on the COC or on the bottles. This must be entered in military time.
- 10.6.7 Client sample ID - Each line is for each individual sample on the COC.
- 10.6.8 The headers IRPMS LOCID (Location), IRPMS SBD (Beginning Depth) and IRPMS SED (Ending Depth) are used only for those clients that place these on the COC.
- 10.6.9 If there is an APPL Trip Blank type in APPL under MS/MSD header. Also if a client designates an MS/MSD this also gets typed in under this header.
- 10.6.10 For the trip blank enter the sampling date and time listed on the container. If there is no sampling date type in 080888; this defaults to NA. If there is no sampling time type in 000000; this defaults to NA.
- 10.6.11 Under the header Additional Tests you would type in those analytes subbed out to another lab, or special requested analytes.
- 10.6.12 Type in the Client Job - This is usually located on the top portion of the COC. The client job description may include a project name, project number or a job name. If a project description is not found then enter the name of the city where the project is located.
- 10.6.13 Make sure the PO number matches what is on the COC. If there is no PO number, then put NA in the cell. If there are more than three PO numbers type them in the comment section instead and type "See comments" in this section.
- 10.6.14 Under the header project manager verify the project managers name is entered correctly.
- 10.6.15 Verify that the Date received is entered correctly.
- 10.6.16 Enter Time Received as the time the samples were received by the lab.
- 10.6.17 Type in the color of all of the refrigerators you are placing the samples into.
- 10.6.18 Under the metals and Wetlab headers enter the individual metals or individual Wetlab tests.
- 10.6.19 To list the analytes requested for each individual sample; go up to Client Name, line 1, right click and choose Edit Test List. Choose the tests to be performed for the first sample on the COC. Double click on the tests available to move them to the right column. When a sample is to be subbed out, double click on SUBB. Add the containers for each sample by choosing @W01, @S01, or @M01. If there are more than one container per sample then you would designate them @W02, @W03, etc. Select save.
- 10.6.20 Go back to Client Name, line 1, right click and choose Edit Special Info. Make sure that every cell except for "Invoice attention to", is filled in correctly. Choose save. For entering the ice chest temperature degree sign hold down the ALT key and type 248.
- 10.6.21 Go back to Client Name, line 1, and right click and choose Edit Comments. Note any comments that are pertinent, such as missed hold times, broken bottles, special analyte lists, air bubbles larger than a pea, client requests a copy of the COC or ARF, PO numbers etc. Select save.



10.6.22 If there is more than one sample then highlight the first line, copy and paste onto the next line and every line after that as needed for the number of samples, as per the COC. Enter the correct date and time of sampling for each sample. Enter the correct client ID exactly as it is written on the COC. If there are different tests for each sample then right click and choose Edit Test list and choose the tests requested on COC for that particular sample. Delete tests not required by double clicking on that test. If there is a special analyte requested or a sample is subbed out type under Additional Tests the analyte and/or subbed analyses.

10.6.23 Select Login on the lower right hand corner.

### 11 Assigning an ARF number

11.1 Obtain the yellow sheet with ARF numbers printed on it. Computer personnel periodically print a yellow sheet with consecutive numbers to be used to assign the ARF number.

11.2 On the next uncrossed out number write in the client's name and cross out the number to show that it has been used.

11.3 Type in the ARF number on the window and select OK.

11.4 The Labworks Sample Container Custody Initialization window comes up. This is a list of the samples you entered along with the bottle designations running across the top (@W01, etc.). Change the white cells to black for all the samples that will go into the refrigerator by clicking on the cell. Click on Assign Location and choose the location, click OK. The refrigerator you have chosen will replace the black cells you made previously. Highlight any other cells that are white, such as all the VOA containers and assign them to the VOA refrigerator in the same way. Save Locations.

11.5 The Labworks desktop sample login window shows up and tells you how many samples are associated with your ARF number. Click OK.

11.6 Minimize the multi log in window.

### 12 Generating the ARF, labels and Inventory Sheet

12.1 On the Labworks desktop double click on Zip Report. Double click on ARF Print.cef.

12.2 In the bottom left corner select ARF number. Select Load.

12.3 Typically the most recently created ARF appears at or near the top of the list. Double click on the desired ARF. If the desired ARF number does not appear in the top of the list then choose Sort by ARF Number and type in the desired ARF number. Choose Okay.

12.4 All the numbers associated with the ARF should show up on the window. Choose Done.

12.5 On the next screen choose Print ARF.

12.6 The ARF will be displayed on the screen. Print it on white paper. Final ARFs with normal turn around times are printed on green paper. Rush ARFs are printed on pink paper. Close this screen and choose Print Labels.

12.7 The next screen will have all the containers received listed on the left side. On the right side will be listed the refrigerator color. To complete the containers inventory sheet highlight the assigned containers (i.e. W01, etc.) and select the pull down menu at the top left corner. Select the appropriate container



description. Select Assign Containers on the upper right side. Continue in this manner until all the containers have a description. Choose print container inventory.

- 12.8 Choose Print Labels for Every Sample. It will ask you if you want to print every sample on the ARF. Choose OK. Exit the window. Place these labels on the containers starting with the VOA containers first, then the next largest container until you reach the largest container. If one of the VOA containers comes in with an air bubble, then do not label that one with W01. Instead make it the third one in line, e.g. W03. Some clients assign a unique number to each container. In these cases arrange the containers in sequential order and then attach the labels. The labels must be placed on the container in the vertical direction and as flat as possible. For odd shaped containers the label may be folded around the container however the section of the label containing the bar code must be kept flat.
- 12.9 APPL sample numbers are placed on the top all containers except VOA containers using the pricing gun. A colored line is drawn above the number on the label, with a marker, corresponding to the color of the refrigerator or freezer where the samples are to be stored.
- 12.10 Other stickers may be placed on the samples when necessary. When a limited amount of sample is received a limited volume sticker is placed on the sample and a notation made on the cooler receipt form. If samples are to be composited then a composite sticker is placed on each container and the number in the sequence indicated. For example: composite 1 of 3, composite 2 of 3 and so on. If the sample is a soil in a brass sleeve or jar and only one container is received for analyses in VOA and the GC or LC/MS section then the container must have a yellow VOA sticker placed on it for the BTEX/Gas analysis or an orange VOA sticker for the 8260B analysis. If the sample is known to be very high in any parameter, a blue sticker is placed on it.
- 12.11 Scan the COC into the T:/drive: Using the copier in receiving press the template button, 007COC, Landscape file, Scan to file, Name. Type in COC + ARF number to name the file, then press scan.

### 13 Completion of the Sample Receiving Quality Control Sign off Sheet

- 13.1 After numbers have been placed on the sample containers and the cooler receipt form and inventory sheet have been completed, the work is peer reviewed. This is documented on the Sample Receiving Quality Control Sign Off sheet (SRQCS) (See Appendix D). The physical portion of the form is completed by someone other than the personnel applying the labels. The ARF section is completed by someone other than the personnel entering the info into the computer.
- 13.2 The communication section of the SRQCS is completed if any of the following occur:
  - 13.2.1 When samples need to be filtered or preserved by the laboratory, the appropriate section is notified in person or by intercom. The sample containers are flagged with a "red glow" sticker.
  - 13.2.2 If the pH of a preserved sample is >2 the appropriate section is notified.
  - 13.2.3 If the sample is rush or has a short holding time or a short amount of holding time remaining (i.e. only 2 days left of a 7-day holding time) the appropriate section is notified.



13.3 The samples are placed in the assigned refrigerator/freezer or secured storage facility. Water samples received for volatile analyses are taken directly to the VOA section. The VOA section places their initials and the time on the form showing they have taken receipt of the samples. When a sample is received that is a hazard or may contaminate other samples, the project manager is notified immediately. The project manager will instruct the sample custodian where to store the sample.

#### 14 Assembling the final ARF

14.1 During the process of completing the paper work any errors found are noted on the white copy of the ARF. All the discrepancies are noted in the comment section, corrections or omission are made, and the ARF is amended (see section 13.0 below). The final ARF and corresponding paperwork is assembled in the following order:

- ARF
- Container Inventory Sheet
- Incoming Sample Notice and/or Client Compound List and/or FAX from client.
- Chain of Custody
- Purchase order form
- Cooler Receipt Form
- Sample Receiving Quality Control Sign Off Sheet (SRQCSO) (Appendix D)

#### 15 Amending the ARF

15.1 Choose Modify

15.2 Choose ARF number and highlight the ARF to be modified, then OK.

15.3 Choose Modify Sample, multisample Spreadsheet.

15.4 There are five windows in the following order: Sample header info, Sample comments, Sample special info, Analysis order, and Setup header editing.

15.5 Go to the following tab or window for editing: Sample Header info

- client information
- job information
- chain of custody number
- turn around time
- submittal information
- refrigerator color
- name of project manager
- invoicing info (in Invoice Address lines 4 & 5 and Report Address line 4)
- sample information
- MS/MSD-APPL-Comp (in Report Address Line 5)
- add test
- metals list
- Wetlab list

15.6 Sample Comments: When you choose sample comments a window will come up showing the list of samples. Choose the first sample in the list. Add any comment needed and enter your initials and the date. No more than six lines can be entered. Choose Save.

15.7 Sample Special Info:



- COC (Y or N)
- RAD screen
- pH
- Custody seals
- Chest temp
- QC report type
- Invoice (first line)

### 15.8 Analysis Order:

15.8.1 In order to delete one or more of the analyses, perform the following step: Under the sample number and to the right of the code you wish to change, right click on Pending. Click on Remove analysis.

15.8.2 To add an analysis, click the first available line under Analysis Ordered. The analysis selection window opens. Either type in the analysis you want to add or scroll down and highlight it. Click OK.

15.8.3 The code will be transferred to your analysis ordered window.

15.8.4 In order to add an analysis to a sample, click on the cell directly across the added code, under the sample number. Test added will show up.

15.9 Setup Header Editing: Remove, add and rearrange headers (columns) as displayed in the Sample Header Info window.

15.10 When all changes are completed, click on Sample Header Info tab located on the bottom left-hand corner. Click Save.

15.11 After any amending is completed, copy the ARF on Green paper for standard TAT, and pink paper for rushes.

15.12 The ARFs are given to reporting department for approval. Any errors or omissions are noted directly on the ARF. If there are changes that need to be made, then amend the ARF as above. Then reprint the ARF on green or pink paper and have the Project Manager sign off on the changes. The ARF is then returned to receiving for distribution.

### 16 Photocopying and Distribution of the ARF

16.1 When the ARF has been approved it is ready to be photocopied and distributed to each applicable section.

16.2 Initial the Copies Made section of the SRQCS. One for each section except for GC/MS, if methods 8260, 524 or 624 are requested a copy goes to VOA. If methods 8270, 525, or 625 are requested then one goes to GC and one to Extraction.

16.3 Make sure all the project managers have initialed all the original ARFs.

16.4 Write the date and time copied next to the printed date and time at the bottom of the ARF.

16.5 Separate multiple copies of the COC. Only one needs to be copied.

16.6 Place the pages on the copier face up.

16.7 Press Sort, front staple,

16.8 Press 1⇒2, the number of copies needed and press print button.

16.9 All original paperwork is attached to the green or pink copy of the ARF and taken to the crates in the office area.

16.10 The copies of the paperwork are stapled and sorted by sections and placed in the corresponding hanging files located in the hallway outside sample receiving.



**17 Making changes to a final ARF**

- 17.1 This is performed when the following circumstances have occurred:
- 17.2 There is an error in logging in the sample analysis method number into LIMS system and the error is not caught during peer review.
- 17.3 There is an error in logging in the date or time of arrival after the ARF is complete. You must obtain the original ARF and make the necessary corrections.
- 17.4 Adding or deleting analysis as per client or project manager.
- 17.5 Change in turn-around-time by client or project manager.
- 17.6 Canceling all analyses by client or project manager. When this occurs obtain the printed ARF in the crate and write cancel, along with your initials and date. See section "Q" for further instructions.
- 17.7 To make any changes to an ARF go to section "M" and make the appropriate changes. All changes are noted in the comment section of the ARF with the date and initials. The ARF is printed on blue paper, signed off by the project manager, then distributed to the appropriate departments.
- 17.8 Any changes that are requested by the client must be documented and the documentation must be attached to the ARF. The documentation may be an amended COC, and email approving changes to the COC or a telephone request to the project manager who makes the changes on the COC, initials, dates and notes the client's name who authorized the change. A copy of the amended COC is sent to the client with the final report.

**18 Re-receiving samples**

- 18.1 If a request for additional analyses or re-analysis is received the information is given to the sample custodian, or his or her designee in writing. Receiving will re-receive the sample(s) in the following fashion.
- 18.2 The sample is pulled and checked to see if sufficient volume in the proper container with the correct preservation is remaining for the additional analyses being requested. The original ARF is pulled and copies are made of the Chain of Custody, Cooler Receipt Form, and Incoming Sample Notice, Client's Compound List or any other information the chemist should have.
- 18.3 The samples are logged into the LIMS system as described in section "F". In the delivered by section enter "in-house". In the comments section enter "These are in-house samples from ARF#...". The original APPL IDs are removed from the containers and the new ones are placed on them. In the COC database the old sample containers are moved to reanalysis.
- 18.4 After numbers have been placed on the sample containers, the work is peer reviewed, the samples are placed back into the assigned refrigerator/freezer.

**19 Canceling an analysis**

- 19.1 When a client requests canceling all analyses sent in before the ARF is prepared, then write Cancel on the COC with your initials and date and file it in the filing cabinets, in the filing room, under the client's name. The samples are then placed on the disposal cart, or sent back to the client, if requested. The samples must be scanned out of the COC database as cancelled.
- 19.2 When client requests canceling all analyses sent in after the ARF is completed, the ARF is modified as described in section "M". After deleting all of the analysis codes you will replace them with the analysis code "cancel". Print the revised ARF on blue paper, have the project manager sign off, and distribute it to the appropriate labs.



# Standard Operating Procedure

## QA Control Copy # 2

SOP: SHR001  
Section: 5  
Revision: 31  
Date: 11/11/09

### SALUTATION

This procedure is applicable to the receiving section personnel.

APPL Inc.  
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Section Manager: \_\_\_\_\_

Date: 11/12/09

QAU Director: \_\_\_\_\_

Date: 11/12/09



APPENDIX A

COOLER RECEIPT FORM

- 1) Project: \_\_\_\_\_ Date Received: \_\_\_\_\_
- 2) Coolers: Number of Coolers: \_\_\_\_\_
- 3) YES NO Were coolers and samples screened for radioactivity?
- 4) YES NO Were custody seals on outside of cooler? How many? \_\_\_\_\_ Date on seal? \_\_\_\_\_
- 5) Name on seal? \_\_\_\_\_
- 6) YES NO NA Were custody seals unbroken and intact at the time of arrival?
- 7) YES NO Did the cooler come with a shipping slip (air bill, etc.)? Carrier name \_\_\_\_\_
- 8) Shipping slip numbers: 1) \_\_\_\_\_ 2) \_\_\_\_\_ 3) \_\_\_\_\_
- 9) YES NO NA Was the shipping slip scanned into the database?
- 10) YES NO NA If cooler belongs to APPL, has it been logged into the ice chest database?
- 11) Describe type of packing in cooler (bubble wrap, popcorn, type of ice, etc.): \_\_\_\_\_

- 12) YES NO NA For hand delivered samples was sufficient ice present to start the cooling process?
- 13) YES NO Was a temperature blank included in the cooler?
- 14) Serial number of certified NIST thermometer used: \_\_\_\_\_ Correction factor: \_\_\_\_\_
- 15) Cooler temp(s): 1) \_\_\_\_\_ 2) \_\_\_\_\_ 3) \_\_\_\_\_ 4) \_\_\_\_\_ 5) \_\_\_\_\_ 6) \_\_\_\_\_ 7) \_\_\_\_\_ 8) \_\_\_\_\_

Chain of custody:

- 16) YES NO Was a chain of custody received?
- 17) YES NO Were the custody papers signed in the appropriate places?
- 18) YES NO Was the project identifiable from custody papers?
- 19) YES NO Did the chain of custody include date and time of sampling?
- 20) YES NO Is location where sample was taken listed on the chain of custody?

Sample Labels:

- 21) YES NO Were container labels in good condition?
- 22) YES NO Was the client ID on the label?
- 23) YES NO Was the date of sampling on the label?
- 24) YES NO Was the time of sampling on the label?
- 25) YES NO Did all container labels agree with custody papers?

Sample Containers:

- 26) YES NO Were all containers sealed in separate bags?
- 27) YES NO Did all containers arrive unbroken?
- 28) YES NO Was there any leakage from samples?
- 29) YES NO Were any of the lids cracked or broken?
- 30) YES NO Were correct containers used for the tests indicated?
- 31) YES NO Was a sufficient amount of sample sent for tests indicated?
- 32) YES NO NA Were bubbles present in volatile samples? If yes, the following were received with air bubbles:  
Larger than a pea: \_\_\_\_\_  
Smaller than a pea: \_\_\_\_\_

Preservation & Hold time:

- 33) YES NO NA Was a sufficient amount of holding time remaining to analyze the samples?
- 34) YES NO NA Do the sample containers contain the same preservative as what is stated on the COC?
- 35) YES NO NA Was the pH taken of all non-VOA preserved samples and written on the sample container?
- 36) YES NO NA Was the pH of acid preserved non-VOA samples < 2 & sodium hydroxide preserved samples > 10?
- Lab notified if pH was not adequate: \_\_\_\_\_

Deficiencies:

Signature of personnel receiving samples: \_\_\_\_\_ Second reviewer: \_\_\_\_\_  
 Signature of project manager notified: \_\_\_\_\_ Date and Time of notification: \_\_\_\_\_  
 Name of client notified: \_\_\_\_\_ Date and Time of notification: \_\_\_\_\_  
 Information given to client: \_\_\_\_\_ by whom (Initials): \_\_\_\_\_



**APPENDIX B**

Sample Acceptance Policy

Please be aware APPL, Inc. has adopted a new sample acceptance policy to insure the integrity of our project and to uphold regulatory requirements. All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria will be noted in the laboratory report defining the nature and substance of the variation.

- ❖ Samples must arrive with labels intact and with a Chain of Custody filled out completely and legibly. The following information must be recorded:
  - Client name, address, phone number and fax number (if available)
  - **Project name and/or number**
  - The sample identification
  - Date, time and location of sampling
  - The collectors name
  - The matrix description
  - **The container description**
  - **The total number of each type of container**
  - Preservatives used
  - Analysis requested
  - Requested turnaround time (TAT)
  - Any special instructions
  - Purchase Order number or billing information (e.g. quote number) if available.
  - The date and time that each person received or relinquished the sample(s), including their signed name as well as the date and time of receipt of when the APPL technician who received the samples in the lab.
  - **Information must be legible**
- ❖ Samples must be properly labeled.
  - Use durable labels (labels provided by APPL, Inc. are preferred)
  - Include a unique identification number
  - Include sampling date and time and sampler ID
  - Include preservative used
  - **Use indelible Ink**
  - **Information must be legible**
- ❖ Proper sample containers must be used that provide adequate volume for the analysis and necessary QC that are required for each analysis requested. APPL, Inc. will provide containers when requested.



- ❖ Samples must be preserved according to the requirements of the requested analytical method or per client QAPP. This includes samples (other than water samples for metals analysis) begin chilling to below 6°C. Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice). A temperature blank must be included in each cooler.

**Chemical preservation (pH) will be verified prior to analysis during login and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation. In the case of volatile analysis, the pH will be taken after analysis.**

- ❖ **Sample Holding Times**

APPL, Inc. will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for samples with short holding time (<48hr HT) samples must be received with at least 60 hrs (2.5 working days) remaining on the holding time for us to ensure analysis.

Analyses that are "field" analyses (e.g. pH, DO, residual chlorine) will be analyzed within 24 hours from receipt of the samples in the laboratory. Field analyses samples received after 4:00pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday).

- ❖ The project manager will be notified if any sample is received in damaged condition. APPL, Inc. will request that a sample be resubmitted for analysis.
- ❖ Recommended for packing samples for shipment:
  - Pack samples in ice rather than "Blue" ice packs.
  - Place ice in zip lock bags to avoid leakage.
  - Soil sample should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
  - Water samples would be best if wrapped with bubble-wrap or paper (newspaper or paper towels work) and then placed in plastic zip-lock bags.
  - Fill extra cooler space with bubble wrap and popcorn.
  - Always include a temperature blank in each cooler.

**I have read and understand the above requirements regarding the sample acceptance policy at APPL, Inc.**

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date



APPENDIX C

INVENTORY SHEET (APPL Sample Receipt Form)

Initials \_\_\_\_\_ Date \_\_\_\_\_

APPL Sample Receipt Form

ARF# 58262

Sample	Container Type	Count	pH
AX91850	<sup>1</sup> PL Liter	1	
	<sup>6</sup> PL 500mL - HNO3	1	1.7
	<sup>13</sup> VOAs - HCL	3	
AX91851	<sup>13</sup> VOAs - HCL	3	

Sample Container Type Count pH

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# Standard Operating Procedure

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SOP: SHR001  
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### APPENDIX D

### SRQCS

Sample Receiving Quality Control Sign Off									
AIR Number:	Analysis Request Form	Reviewer's Initials/Date	Physical Check of Samples	Reviewer's Initials/Date					
	Is the chain of custody attached?	Yes	APPL ID number is placed on correct samples?	Yes	No	NA			NA
	Is the QC number entered correctly?	Yes	Color of refrigeration correct on labels?	Yes	No	NA			NA
	Is RAD scanner performed entered correctly?	Yes	Is inventory sheet been completed correctly?	Yes	No	NA			NA
	Is pH check performed entered correctly?	Yes	Are pH's recorded on samples?	Yes	No	NA			NA
	Is 1st round line entered correctly?	Yes	Are pH's equilibrated?	Yes	No	NA			NA
	Do labels received enter correctly?	Yes	Labels placed on samples for VOA, GC, GC/MS?	Yes	No	NA			NA
	Time received entered correctly?	Yes	Water level, composite, or initial volume etc are entered?	Yes	No	NA			NA
	Delivered by name entered correctly?	Yes	Is water present in the 30l yes the container put in fig?	Yes	No	NA			NA
	Is shuttle cushion seals entered correctly?	Yes	Water 16-21, 2, A-1, 1, DM, 2, 1, 1, 1 snails placed in the fig?	Yes	No	NA			NA
	Is water level entered correctly?	Yes	Communication		No	NA			NA
	Project number entered correctly?	Yes	Sections notice of wet samples or about holding times?	Initials:	No	NA			NA
	Client ID's from chain entered correctly?	Yes	Sections notice if filtering was needed?	Yes	No	NA			NA
	APPL ID's save correct matrix designation?	Yes	Sections notice in adjusted pH?	Yes	No	NA			NA
	Sample date entered correctly?	Yes		Trax:	No	NA			NA
	Sample line entered correctly?	Yes	Receipt of samples by VDAMS		No	NA			NA
	MSMSD requests indicated?	Yes			No	NA			NA
	Signature indicated?	Yes	Distribution of AIR		No	NA			NA
	Limit volume indicated?	Yes			No	NA			NA
	Air samples in hold entered correctly?	Yes			No	NA			NA
	Air submittal CDCs attached?	Yes			No	NA			NA
	QC scanned to pdf?	Yes			No	NA			NA
					No	NA			NA
	Project Manager Initials/Date				No	NA			NA
	Client name correct?	Yes	Dept 1 sth	MS/DNA	No	NA			NA
	Client address correct?	Yes	GC	Initials	No	NA			NA
	Attention name correct?	Yes	Ext action Lab	LCMS	No	NA			NA
	Client phone number and fax number correct?	Yes		Receiving	No	NA			NA
	Project name or number correct?	Yes		Initials/Trax:	No	NA			NA
	PC number correct?	Yes			No	NA			NA
	Current air analytes supplied is listed in AIR?	Yes			No	NA			NA
	Correct Labworks codes used for each method?	Yes			No	NA			NA
	Correct codes used for client/project?	Yes			No	NA			NA
	Correct mass listed?	Yes			No	NA			NA
	Submittal CDCs correct?	Yes			No	NA			NA
	QC results listed correctly?	Yes			No	NA			NA
	Comments entered correctly?	Yes			No	NA			NA
	Invoicing information entered from chain?	Yes			No	NA			NA
	A special spike, if needed, is entered?	Yes			No	NA			NA
	Air samples in hold labeled attached?	Yes			No	NA			NA
	Limit volume indicated, if needed?	Yes			No	NA			NA



## Standard Operating Procedure

### SAMPLE DISPOSAL AND WASTE COLLECTION, STORAGE AND DISPOSAL

---

#### STATEMENT OF PURPOSE

This procedure describes the way in which samples are purged and the proper collection and disposal of contaminated wastes.

#### INSTRUCTIONS

##### **Step 1 – Samples Ready for Disposal – Performed by Information Systems personnel**

The progress of the samples, including the mailed and disposal status, is tracked using the ARFsummary database. When the report is sent out, the mailed status is changed to true, the date is entered, and initialed. The sample containers must be held a minimum of 15 days from this date. If the sample is on hold and the matrix is not water, the sample containers are held for a minimum of 45 days. The APPL Disposal Database includes a list of clients who require permission prior to disposal of samples and / or clients who require longer sample storage time than APPL's standard post-analysis storage times. The samples associated with these clients will not appear in the routine disposal data base, but will remain within APPL's inventory of samples until permission has been granted by the client for disposal and / or the client's specific post-analysis storage time has been met. A table is compiled of samples meeting the retention requirements and a disposal status of false. This table includes the sample number, the state of origin, and hazard status. The sample results are used to determine if there are any analytes that exceed hazardous waste limits. The lists of hazardous waste limits are attached to this SOP. Any analyte exceeding the hazardous waste limit is classified as metal, pesticide, or VOA and entered into the hazard status field as M, P or V, respectively. The table is uploaded to a cordless barcode scanner used to purge the refrigerators.

To accomplish Step 1 using the Disposal database:

1. Insure that the 'Automate' button is checked and click 'Put Scanner in Host Mode'
2. You can probably skip this step. The automation process will go past it. If you need to do it, read below. But the reasoning for not having to is given below as well: Click "Update Disposal Hits". (Note: this process takes hours and can be skipped if the date on the form titled 'Data Updated' is a recent date. This process attempts to automate itself daily at 4am so it is ready by opening hours. That date will not change daily as at any given time, 15 days in the past falls on weekends. Thus there is no point on doing this step on Tuesday and Wednesday as no samples go out on the weekend. It is advised to skip this step if the 'Data Updated' date is recent!)
3. Step 3 will automatically initiate itself. If not, click 'Check Disposal Limits'. Sort tblDisposalLimits by "Limit" to see if any are missing. Have Leonard or Jeremy fill in any missing limits. If there were any missing, close the table.
4. If Step 3 had missing limits and you've fixed them, Click "Export Disposal Samples". If step 3 had no missing limits, this step will initiate itself.
5. Step 5 will initiate itself. If not, click 'Open MCL-Link'.
6. Step 6 Compact database – this is grayed out and may be skipped.
7. Step 7 will initiate itself. If not, click 'Disposal Location'.



8. You are now done on the database side, time to update the scanner. Make sure that the scanner is in host mode. From system menu<sup>1</sup>, 1, "ENTER" (4 times)
9. From the MCL-Link program that you opened in step 5, Click 'Send' and then the green checkmark for Send B.dat to B.dat
10. Reboot scanner. FUNC, \*
11. Click "Disposal Location", print out report (Lists locations needed to be purged)

### **Step 2 – Purging the Refrigerators – Performed by Receiving personnel**

Each sample container has a label with a sample number barcode and a container barcode OR a single barcode containing both the sample number and container ID in one barcode. The sample number barcode is scanned. If the sample number is not in the table, the scanner prompts for another sample number. If the sample number is in the table, the scanner prompts for the container and the container barcode is scanned. (In the case that the sample number and container ID are combined into one barcode, it will not ask for the container ID as it has already retrieved it.) The scanner displays the state of origin and the hazard status. The scanner prompts the disposal location. The choices are printed with barcodes including "Garage", "Drain", "Dumpster", "CANCEL", and "Baked". The disposal location is determined in the following order. If the hazard status contains M, P, or V, the user needs to scan "Garage". If the matrix is water, the user needs to scan "Drain". If the state of origin is "CA", the user needs to scan "Dumpster". Otherwise, the user needs to scan "Baked". If a mistake is made, such as a Hazardous sample is scanned for DRAIN, then the sample must be rescanned and the location to be chosen is CANCEL. Thus the COC database will never show the sample as being moved due to the error. If needed, the sample can be scanned a third time to move the sample to its correct location. The user puts the sample container with others going to the same disposal location. The scanner prompts for another sample number.

### **Step 3 – Update Chain Of Custody Database – Performed by Information Systems personnel**

Sample containers are tracked throughout the laboratory using the Chain of Custody database (COC DB). Each time a container is moved to a new location, an entry is added to the COC DB. When a disposal location is assigned, the sample number, container, location, user, date, and time are entered into a table in the scanner. This information is download from the scanner into the Disposal Database. The location is verified against the hazard status, matrix, and state of origin as described above. Any duplicate entries are deleted. Hazard labels are printed for any containers moved to "Garage". The new location is added to the COC DB. If the current location of every container for a sample number is in a disposal location, the ARFsummary database is updated so that the disposal status is true.

To accomplish Step 3 using the Disposal database:

8. Ensure that the 'Automate' checkbox is checked and click 'Put Scanner in Host Mode'. Follow the directions printed to the screen
9. Click 'Open MCL-Link' and on the MCL-Link program that opens, click 'Receive' and then the green checkbox for Receive A.dat to A.dat.
10. Click 'Import Inventory Table – MUST get confirmation!!!' and read the message boxes carefully, follow them to a T.
11. Click 'Verify Data'. Check qryInventoryDups for duplicate sample containers. Delete entries from tblP360Inventory as necessary. Check qryInventoryHazard. Make sure

<sup>1</sup> System menu: FUNC, \*, FUNC, BK



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all/only hazards are in "Garage", waters in "Drain", "CA" soil/misc in "Dumpster", and other soil/misc in "Baked". Correct as necessary.

12. Click 'tblP360Inventory to COC DB', this step will take a minute or two.
13. Step 13 will initiate itself. If not, click 'Update ARFsummary from COC DB'. This step will take 4-5 minutes.
14. Step 14 will initiate itself. If not, click 'Print Hazard Labels'. Hazard labels will now come off of the label printer next to the Receiving Manager's desk. Place these hazard labels on the appropriate container destined for the Garage.
15. Step 15 will initiate itself. If not, click 'Update tblDisposedFrig'
16. Step 14 will initiate itself. If not, click 'Delete Scanner Data' and follow the instructions on the screen. The general instructions are: Delete file "A" from scanner. From system menu<sup>2</sup>, 4, MODE, 7, ENTER

### **Step 4 – Hazardous Samples – Performed by Receiving personnel**

Hazard labels are applied to samples with a disposal location of "Garage". The label includes the sample number, container, and the classification of hazards (M, P and/or V). Then samples are sorted by their classification and placed in the garage. When the waste recycler removes the samples, the new location will be entered into the COC DB. Waste which is to be disposed is contracted to a licensed company dealing in hazardous waste hauling & disposal. This includes but may not be limited to contaminated samples, used oil, or spent solvents. A list of containers and their contents is provided to the subcontracting firm to facilitate disposal.

### **Step 5 – Scheduling a Pick up – Performed by Receiving personnel**

To schedule a waste pick up the subcontracting firm will be called to arrange a date and time.

### **SALUTATION**

This procedure is applicable to shipping and receiving personnel.

Section Manager: \_\_\_\_\_

Date: 7/7/09

QAU Director: \_\_\_\_\_

Date: 7/7/09

<sup>2</sup> System menu: FUNC, \*, FUNC, BK



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### Fresno Municipal Local Limits for Water ( $\mu\text{g/L}$ )

Arsenic	320
Cadmium	120
Copper	2500
Cyanide	770
Lead	1200
Mercury	50
Nickel	1300
Silver	1100
Total Chromium	6700
Zinc	2100
pH	6.0-12.4
Phenolic compounds	300,000
TCE	120
PCE	770
BTEX	20,000
Oil & Grease	700,000
Benzene	500

### Water limits not regulated by Fresno County ( $\mu\text{g/L}$ )

2,4-Dichlorophenoxyacid	10,000
2,4,5-TP(Silvex)	1,000
Aldicarb	10,000
Aldrin	1,400
Asbestos	10,000,000
Atrazine	15,000
Baygon	90,000
Bentazon	8,000
Bolero(thiobencarb)	10,000
Bromacil	50,000
Captan	350,000
Carbaryl	60,000
Chlordane	30
Chloropicrin	50,000
Cresol	200,000
o-Cresol	200,000
m-Cresol	200,000
p-Cresol	200,000
DBCP,EDB	100
DDD,DDT,DDE,TDE	500
Dicamba	10,000
Dieldrin	8,000
Dimethoate	14,000
Dioxin	10
Diphenamid	40,000
DNBP	no limit
Endosulfan	1,000
Endrin	20



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### Water limits not regulated by Fresno County (continued) ( $\mu\text{g/L}$ )

Ethion	35,000
Glyphosate	500,000
Gross Alpha	no limit
Heptachlor	8
Kepone	21,000
Lindane	400
Malathion	160,000
Methoxychlor	10,000
Methyl Parathion	30,000
Mirex	21,000
Ordram(Molinate)	20,000
Parathion	30,000
PCB's	50,000
Pentachlorophenol	17,000
Phthalates	100,000
Pyridine	5,000
Simazine	150,000
THC	1,000,000
Toxaphene	5,000
2,4,5-Trichlorophenol	400,000
2,4,6-Trichlorophenol	2,000
Trifluralin	100,000
Trithion	7,000
Ammonia	1,000,000
Antimony	500,000
Arsenic	5,000
Barium	100,000
Beryllium	75,000
Cadmium	1,000
Chloride, Nitrate, Sulfate	no limit
Chromium	5,000
Chromium+6	5,000
Cyanide	1,000
Cobalt	8,000,000
Copper	2,500,000
Fluoride Salts	18,000,000
Lead	1,200
Mercury	200
Molybdenum	3,500,000
Nickel	2,000,000
Oil & Grease	no limit
Selenium	1,000
Silver	5,000
Thallium	5,000
TOX	1,000,000
Vanadium	2,400,000
Zinc	5,000,000



Soil Limits of Contamination ( $\mu\text{g}/\text{kg}$ )

2,4-Dichlorophenoxyacid	200,000
2,4,5-TP (Silvex)	20,000
Aldicarb	200,000
Aldrin	28,000
Asbestos	200,000,000
Atrazine	300,000
Baygon	1,800,000
Bentazon	160,000
Bolero (thiobencarb)	200,000
Bromacil	1,000,000
Captan	7,000,000
Carbaryl	1,200,000
Chlordane	600
Chloropicrin	1,000,000
Cresol	4,000,000
o-Cresol	4,000,000
m-Cresol	4,000,000
p-Cresol	4,000,000
DBCP, EDB	2,000
DDD, DDT, DDE, TDE	10,000
Dicamba	200,000
Dieldrin	160,000
Dimethoate	280,000
Diphenamid	800,000
DNBP	no limit
Endosulfan	20,000
Endrin	400
Ethion	700,000
Glyphosate	10,000,000
Gross Alpha	no limit
Heptachlor	160
Kepone	420,000
Lindane	8,000
Malathion	3,200,000
Methoxychlor	200,000
Methyl parathion	600,000
Mirex	420,000
Ordram (Molinate)	400,000
Parathion	600,000
PCBs	1,000,000
Pentachlorophenol	340,000
Phthalates	2,000,000
Pyridine	100,000
Simazine	3,000,000
THC	20,000,000



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### Soil Limits of Contamination (continued) ( $\mu\text{g}/\text{kg}$ )

Toxaphene	10,000
2,4,5-Trichlorophenol	8,000,000
2,4,6-Trichlorophenol	40,000
Trifluralin	2,000,000
Trithion	140,000
Trichloroethene	1200
Tetrachloroethene	7700
Ammonia	20,000
Antimony	10,000
Arsenic	100
Barium	2,000
Beryllium	1,500
Cadmium	20
Chloride, Nitrate, Sulfate	no limit
Chromium	100
Chromium +6	100
Cyanide	20
Cobalt	160,000
Copper	50,000
Fluoride Salts	360,000
Lead	100
Mercury	4
Molybdenum	70,000
Nickle	40,000
Oil & Grease	no limit
Selenium	20
Silver	100
Thallium	100
TOX	20,000
Vanadium	48,000
Zinc	100,000

### Water or Soil Limits of explosives

As determined by Diane Anderson on 2008.11.14, explosives have the following limits:

mg/kg or mg/L	10
$\mu\text{g}/\text{kg}$ or $\mu\text{g}/\text{L}$	10,000

A.P.P.L., INC.  
CONFIDENTIALQA CONTROL COPY # 4APPL,  
INC.**Standard Operating Procedure****PCDD's and PCDF's (EPA METHOD 8290<sup>1</sup>)  
SOXHLET EXTRACTION of Soil/Sediment****STATEMENT OF PURPOSE**

This procedure describes the Soxhlet extraction for Soil/Sediment samples for Polychlorinated Dibenzodioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) analysis by high-resolution GC/MS. The extraction code for this method is SOX8290S.

**INSTRUCTIONS****1.0 Scope and Application**

- 1.1. This SOP describes a procedure for isolation and concentration of PCDD and PCDFs in solid matrices. This procedure also describes the concentration techniques suitable for preparing the extract for the EPA 8290<sup>1</sup> cleanup or for direct analysis by HR GC/MS if cleanup is not required.
- 1.2. 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.
- 1.3. If an individual project has its own QAPP with client specific requirements that are different than the SOP, the QAPP overrides the SOP. This information will be specified in the comment section of the ARF.

**2.0 Method Summary**

- 2.1. The percent dry weight of soil/sediment samples is first determined.
- 2.2. A measured amount of sample, usually 10g (dry weight) of soil sample, is extracted with the appropriate solvent using Soxhlet extraction.
- 2.3. The extract is macro-concentrated by Rotovap and, as necessary, exchanged into a solvent or column compatible with the cleanup to be used.
- 2.4. The eluent from the final cleanup is micro-concentrated under nitrogen and brought to a final volume of 50 $\mu$ L.

**3.0 Sample Preservation, Containers, Handling and Storage**

- 3.1. Soil samples should be collected in glass jars fitted with Teflon lined lids and stored at 4°C  $\pm$  2 °C until delivery to the laboratory.
- 3.2. When the samples are delivered to the laboratory they are placed into a refrigerator in the sample receiving area that is kept at 4°C  $\pm$  2°C.
- 3.3. Samples must be extracted within 30 days of sampling.
- 3.4. Extracts must be stored at room temperature in the dark. Extracts must be analyzed within 45 days of extraction.

**4.0 Interferences and Potential Problems**

- 4.1. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under conditions of the analysis by

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analyzing method blanks. All solvents used are lot tested for acceptability prior to use.

- 4.2. A 4L container of each solvent lot is received by the laboratory. The lot number is prepared per SOP ORG041 and the extract is given to the appropriate section for the determinative step. If the results of the extract have no target analytes above the MDL, the lot number is accepted.
- 4.3. Phthalates esters contaminate many types of products commonly found in the laboratory. Serious phthalate contamination can occur if consistent quality control is not practiced.

## 5.0 Equipment/Apparatus

- 5.1. Nitrogen blowdown apparatus
- 5.2. Balances capable to accurately weighing to 0.01g and 0.0001g
- 5.3. Centrifuge
- 5.4. Water bath with temperature controlled within  $\pm 2^{\circ}\text{C}$
- 5.5. Glove box
- 5.6. Stainless steel spoons and spatulas
- 5.7. Laboratory hoods
- 5.8. Pipettes, disposable, Pasteur, 150mm long X 5mm ID
- 5.9. Pipettes, disposable, serological, 25mL
- 5.10. Glass injection vials (0.3mL conical)
- 5.11. Mortar and Pestle
- 5.12. Blender
- 5.13. 6mL SPE glass tubes
- 5.14. Glass fiber filters (Whatman 0.70um)
- 5.15. Glass funnels
- 5.16. Desiccator
- 5.17. Rotary evaporator and water bath (with micro-concentration adaptor when needed)
- 5.18. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar
- 5.19. 500mL flat bottomed boiling flasks
- 5.20. Silica beads (100 mesh)
- 5.21. Assorted Class "A" syringes
- 5.22. SPE Vacuum Manifold
- 5.23. Soxhlet extractor apparatus
- 5.24. Soxhlet thimbles
- 5.25. Conical glass centrifuge tube (15ml)
- 5.26. Erlenmeyer Flask (250mL, 1000mL)
- 5.27. Explosion proof hot plate
- 5.28. Aluminum foil
- 5.29. Laboratory oven (capable of sustaining temperatures up to  $400^{\circ}\text{C}$ )
- 5.30. 125ml separatory funnel (for soil back-extraction)
- 5.31. Supelco Custom SPE Cartridges (Carbon/Celite)
- 5.32. Supelco Custom SPE Cartridges (Acid/Base Silica Gel)

## 6.0 Reagents.

- 6.1. Organic free water
- 6.2. Quartz sand (or Ottawa Sand)
- 6.3. Concentrated Sulfuric acid – reagent grade

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- 6.4. 20% Potassium Hydroxide solution - (20g KOH dissolved into 100mL DI Water)
- 6.5. 5% Sodium Chloride solution - (5g NaCl dissolved into 100mL of DI Water)
- 6.6. Methylene chloride - reagent grade
- 6.7. Nonane - reagent grade
- 6.8. Toluene - reagent grade
- 6.9. Hexane - reagent grade
- 6.10. Acetone - reagent grade
- 6.11. Methanol - reagent grade
- 6.12. Cyclohexane - reagent grade
- 6.13. Activated Silica Gel-100 mesh (methylene chloride rinsed and baked @ 180°C for 1 hour: cooled in Desiccator and stored in glass jar with Teflon-screw cap)
- 6.14. Acidic Silica Gel (100g activated silica gel + 44g concentrated H<sub>2</sub>SO<sub>4</sub> mixed well and stored in glass jar with Teflon-screw cap)
- 6.15. Basic Silica Gel (100g activated silica gel + 30g of 1N NaOH mixed well and stored in glass jar with Teflon screw cap)
- 6.16. Celite 545 (stored in a sealed container at room temperature)
- 6.17. Active carbon AX-21 (pre-washed with methanol and dried in vacuum at 110°C. Store in a glass bottle sealed with a Teflon lined screw cap.
- 6.18. Activated Anhydrous Sodium Sulfate (methylene chloride rinsed and baked @ 400°C for 1 hour: cooled in Desiccator and stored in glass jar with Teflon-screw cap)

## 7.0 Procedure:

### 7.1. Determining Percent Dry Weight:

- 7.1.1. Weigh a 10g portion of soil ( $\pm 0.5g$ ) to three significant figures.
- 7.1.2. Dry it to constant weight at 100°C in an adequately ventilated oven.
- 7.1.3. Allow the sample to cool in a desiccator and weigh the dried solid to three significant figures.

$$\% \text{dry weight} = \frac{\text{wt of dry sample}}{\text{wt of sample}} \times 100$$

### 7.2. Preparation for Extraction:

#### 7.2.1. Preparation of Soil Samples:

- 7.2.1.1. Add 10g of anhydrous powdered sodium sulfate to 10g of soil (dry weight) and mix thoroughly.
- 7.2.1.2. Place mixture in Soxhlet apparatus on top of a glass wool plug or Soxhlet thimble and add 250mL of methylene chloride to the flask.
- 7.2.1.3. Spike the samples, blanks, LCS and MS/MSD with the acetone diluted surrogate solution. Spike the LCS and MS/MSD with spike mix.
- 7.2.1.4. Reflux for 16 hours. The solvent must cycle completely through the system five times per hour.

**NOTE:** The addition of spike and surrogate will be witnessed by a second person and will be documented on the extraction sheet.

- 7.2.1.5. Cool and filter through a glass fiber filter into a 500mL round bottom flask.
- 7.2.1.6. Rinse filter with 10mL of methylene chloride and using a Rotovap, concentrate to near dryness at 40°C. Solvent transfer to hexane. Allow to cool.

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7.2.1.7. Transfer the residue to a 125mL separatory funnel with 15mL of hexane and rinse flask with two additional 15mL portions of hexane.

7.2.1.8. Proceed to cleanup stage.

### 7.3 Cleanup Stage

#### 7.3.1 Acid/Base Partition

7.3.1.1 Add 50mL of the H<sub>2</sub>SO<sub>4</sub> solution from sect 6.3. Shake for 2min and discard the aqueous layer. Repeat the "acid washing" until no color is visible in the aqueous layer for a maximum of 4 washings.

7.3.1.2 Add 40mL of NaCl solution from sect 6.5. Shake for 2min and discard the aqueous layer.

7.3.1.3 Add 40mL of 20% potassium hydroxide from sect 6.4. Shake for 2min and discard the aqueous layer. Repeat the "base washing" until no color is visible in the aqueous layer for a maximum of 4 washings.

7.3.1.4 Add 40mL of NaCl solution from sect 6.5. Shake for 2min and discard the aqueous layer.

7.3.1.5 Dry the extract by pouring it through a filter funnel containing anhydrous sodium sulfate on a glass wool plug, and collect it in a Rotovap flask. Rinse the funnel with the sodium sulfate with two 15mL portions of hexane, add the rinses to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (if any) are removed.

7.3.2 Silica Column Cleanup (Supelco Custom Silica Gel SPE Cartridges may be used for this step, or the technician may prepare his own column as follows):

7.3.2.1 Preparation of Silica/Alumina Columns for sample cleanup (as needed). Pack SPE glass tube listed in sect 5.13 with the following materials from bottom to top:

Glass wool plug

Activated silica gel (1.0g) – see sect 6.15

Basic silica gel (2.0 g) – see sect 6.17

Acidic silica gel (4.0g) – see sect 6.16

Activated silica gel (2.0g) – see sect 6.15

7.3.2.1 Elute the column with 10mL hexane. Be careful not to let the column run dry.

7.3.2.2 Dissolve the residue from 7.3.1.5 in 2mL of hexane and apply the hexane solution to the top of the silica gel column. Rinse the flask with enough hexane (3-4mL) to complete transfer of sample. Elute the silica gel column with 90mL of hexane. Concentrate the eluent on a rotary evaporator (35°C water bath) to approximately 1mL.

7.3.3 Carbon Column Cleanup (Supelco Custom Carbon SPE Cartridges may be used for this step, or the technician may prepare his own column as follows):

7.3.3.1 Preparation of an AX-21/Celite 545 column for sample cleanup (as needed): Mix 5.4g active carbon Ax-21 (Sect 6.19) and 62.0g Celite 545 (Sect 6.18). Activate the mixture at 130°C for 6 hours and store it in a desiccator. Using a triangular file, score the ends of a 25mL disposable serological pipette. Carefully snap off the ends and pack with the following materials from bottom to top:

Glass wool plug

1cm plug of Celite 545 (see Sect 6.18)



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1cm plug of the AX-21/Celite 545 Mixture (see Sect 7.2.3.1)  
1cm plug of Celite 545 (see Sect 6.18)  
Glass wool plug

Note: The following elution procedure may be used with laboratory-made carbon columns or the purchased SPE carbon cartridges, without the use of a SPE vacuum manifold.

- 7.3.3.2 Rinse the AX-21/Celite 545 column with 5mL of toluene.
- 7.3.3.3 Rinse with 2mL of (75:20:5, v/v) methylene chloride/methanol/toluene solution.
- 7.3.3.4 Rinse with 1mL of (1:1, v/v) Cyclohexane/methylene chloride solution.
- 7.3.3.5 Rinse with 5mL of hexane.
- 7.3.3.6 The flow rate should be less than 0.5mL/min. Discard the rinses.
- 7.3.3.7 While column is still wet with hexane, add the sample concentrate.
- 7.3.3.8 Rinse the concentrator tube twice with 1mL hexane.
- 7.3.3.9 Rinse column sequentially with two 2mL portions of hexane, 2mL Cyclohexane/methylene chloride (50:50, v/v), and 2mL-methylene chloride/methanol/toluene (75:20:5, v/v). Combine these eluents: This combined fraction may be used as a check on column efficiency.
- 7.3.3.10 Turn the column upside down and elute the PCDD/PCDF fraction with 20mL of toluene. Add the rinse to the eluent.
- 7.3.3.11 Concentrate the extract in a rotary evaporator (50°C water bath), to near dryness. Add 1mL of hexane.
- 7.3.4 Micro-Concentration: Quantitatively transfer the sample extract from the boiling flask to a 15mL centrifuge tube. Rinse out flask with two 5mL portions of hexane and add it to the centrifuge tube. Assemble the extracts in a nitrogen blow-down apparatus.
  - 7.3.4.1 Adjust the flow of the Nitrogen gas until the surface of the solvent is just visibly disturbed. When the volume of the eluent has concentrated to approximately 100µL, transfer the concentrate extract into a 300µL conical injection vial (marked at the 50µL level) for further concentration. Rinse centrifuge tube three times with 300µL of hexane. Between rinses, continue concentrating to 100µL volume and transfer concentrate to injection vial.
  - 7.3.4.2 Add 30µL of nonane and 20µL Internal Standard in nonane to the extract and continue concentrating under nitrogen until the 50µL level is reached. The sample is now ready for instrument analysis (see SOP ANA8290).

8.0 Calculations – NA

9.0 Quality Control

- 9.1 Demonstration of Capability (DOC) – You must demonstrate initial proficiency by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This consists of preparing four lab controls with a second source standard, or spike prepared independently of the calibration. The DOC must also be performed for each of the cleanup steps listed in section 7.4.
- 9.2 To all samples, blanks, laboratory control spikes (LCS) and matrix spikes (MS/MSD) add the surrogate solution and spiking solution as listed on the extraction sheet.

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- 9.3 The Organic Extraction supervisor, the Section Manager, or a properly trained analyst will perform surrogate/spike additions.
- 9.4 Each batch of no more than 20 samples should include a Laboratory Control Sample (LCS) and a Laboratory Method Blank. These should consist of DI water and be extracted just like the samples.
- 9.5 Each batch should also include either a Matrix spike and an unspiked sample duplicate or a matrix spike and a matrix spike duplicate (MS/MSD) if the client has provided enough sample volume.

## 10.0 Data Validation - NA

### 11.0 Pollution Prevention

All hazardous materials that are generated during the testing of samples must be properly collected and stored. Drums are available in the storage room for the following types of wastes- acidic, basic and solvents.

### 12.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases from fume hoods and bench operations.

### 13.0 Contingencies for Handling Out of Control or Unacceptable Data

In the event that an out of control situation occurs, the project manager will be notified immediately. The affect of the out of control situation will be assessed according to the project DQO. If sufficient sample remains, and the situation will significantly affect the quality of the results, the analysis will be repeated. If the situation does not significantly affect the quality of the data, the project manager will notify the client and instructions from the client will be followed. In the event no sample remains, the client will be notified immediately. All situations will be documented on the multi level sheet and initialed by the project manager. All out of control situations will be brought to the attention of the QAU in the form of a QCER. The QAU has the final authority to approve the actions taken.

### 14.0 Deviations to the method

This SOP was compared to method 8290. There are no deviations to the method.

### 15.0 Health and Safety

Lab coats safety glasses and gloves are used at all times. A face shield will be worn when handling glass separatory funnels. Some samples require the use of respirators. This is on a case by case basis.



Standard Operating Procedure

APPL, INC.

SOP: SOX8290S  
Section: 6  
Revision: 1  
Date: 01/19/09

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CALCULATION

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This procedure is applicable to all personnel who perform separatory funnel extractions on water samples for EPA Method 8290<sup>1</sup> analysis.

Section Manager: *De Gr*

Date: 1/21/09

QAU Director: *Francis Pedraza*

Date: 1/21/09

<sup>1</sup> USEPA Method 8290, Revision 0, September 1994. Polychlorinated Dibenzodioxins and Polychlorinated Dibenzodioxins by HRGCMS



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Table 1

Dioxin/Furan Soil/Sediment Extraction Flow Chart

SOIL/SEDIMENT SAMPLES      SOP Sect

Determine percent dry weight	7.1
Soxhlet preparation	7.2.1.1
Add Spike and Surrogate	7.1.1.3
Reflux for 16 hours	7.2.1.4
Rotovap to near dryness	7.1.1.5
Transfer to separatory funnel with hexane	7.1.1.6
Acid Partition	7.3.1.1
NaCl Partition	7.3.1.2
Base Partition	7.2.1.3
NaCl Partition	7.2.1.4
Rotovap to near dryness	7.2.1.5
Silica Gel Cleanup	7.2.1.5
Rotovap to 1mL	7.2.2.3
Carbon Cleanup	7.2.2.4
Rotovap to 1mL	7.2.3.7
Conc. under N <sub>2</sub> (25µL I.S. in Nonane)	7.2.3.11



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Table 2

"Spike" and "Surrogate" solutions

**Dioxin/Furan "Spike"**

20 $\mu$ L of CIL EDF-5008 spiked to a final volume of 20mL with ACETONE solvent

(Add 1.0mL of the Dioxin/Furan Spike Mix to each 1.0 Liter aliquot of LCS, MS/MSD)

**Dioxin/Furan "Surrogate"**

25 $\mu$ L CIL EDF-5005 spiked into each 1.0 Liter aliquot of Blank, LCS, MS/MSD, and SAMPLE

(Add 25 $\mu$ L of the Dioxin/Furan Surrogate to each 1.0 Liter aliquot of LCS, MS/MSD)

**Dioxin/Furan "Internal Standard"**

20 $\mu$ L of CIL EDF-4055 in Nonane solvent

(Add 20 $\mu$ L of the I.S. to each 100 $\mu$ L extract prior to micro-concentration of the Blank, LCS, MS/MSD and SAMPLE)

*Note 1: See APPL SOP ANA8290 for a list of the Dioxin/Furans present in each mix shown above.*

**METALS DIGESTION/PREPARATION**

**METHODS**

**USEPA SW846**

**3005A, 3010A, 3030C, 3031, 3050B**

**USEPA CLPILM 04.1 Aqueous & Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June, 2003)**

**Addendum for USEPA CLPILM 05.2 Aqueous & Soil/Sediment**

**USEPA Methods for Chemical Analysis of Water and Wastes**

**200.7, Standard Methods 3030C**

**SOP NUMBER:**

**SOP-100**

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**07/25/06**

**EFFECTIVE DATE**

**04/20/09**

**DATE OF LAST REVIEW**

## METALS DIGESTION/PREPARATION

### References:

**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3031, 3050B**

**USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C**

**See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

## I. SCOPE AND APPLICATION

### A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
  - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
  - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
  - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
  - a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

**B. SOLIDS**

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
  - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
  - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
  - a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

**C. OILS**

1. Method 3031, "Digestion Procedure for Oils".
  - a. This method is used to prepare samples containing oils, greases or waxes for analysis by inductively coupled argon plasma emission spectroscopy (ICP).

**D. NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

**II. SUMMARY OF METHODS**

- A. A representative sample of water, soil or oil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration must be used in order to reach that lower level. Care

must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike ( not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

### **III. SAMPLE HANDLING AND PRESERVATION**

#### **A. AQUEOUS**

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO<sub>3</sub> to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO<sub>3</sub> is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

#### **B. SOLIDS**

1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

#### **C. OILS**

1. Samples are taken in high density polyethylene bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

### **IV. INTERFERENCES**

#### **A. AQUEOUS**

1. Method 3005A and USEPA CLPILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy", SW846, July, 1992.
  - a. This digestion procedure may not be sufficiently vigorous to destroy some metal complexes.
2. Method 200.7

3. Method 3010A
  - a. See method 6010B.

## B. SOLIDS

1. Method 3050B
  - a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.
2. USEPA CLP ILM0 4.1
  - a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

## C. OILS

1. Method 3031
  - a. These digestates can have very high dissolved solids, which may necessitate the use of internal standards, dilutions, or the method of standard addition.

## V. SAFETY

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
- B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
- C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

## VI. EQUIPMENT/APPARATUS

- A. Fume hood, Labconco or equivalent.

- B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.
- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
- D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
- E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
- F. Beckman CS-6R centrifuge.
- G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
- H. Whatman No. 41 filter paper or equivalent.
- I. Whatman No. 42 filter paper or equivalent.
- J. Whatman 0.45 micron filter paper or equivalent.
- K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
- L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
- M. Manual Sample Mill
- N. Wiley Sample Mill
- O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

## **VII. REAGENTS AND STANDARD PREPARATION**

### **A. REAGENTS**

1. Metals grade Nitric acid ( $\text{HNO}_3$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid ( $\text{HCl}$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid ( $\text{H}_2\text{SO}_4$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
9. Base oil, analyte-free. Oil should be analyzed to determine level of impurities. If method blank is < MDL, then the reagent can be used.

## **B. STANDARDS**

### **1. Traceability**

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

## 2. PREPARATION

### A. Laboratory control sample

#### 1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO<sub>3</sub>, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIIM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO<sub>3</sub>, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO<sub>3</sub> and 5% HCl. Use 0.125 mLs HNO<sub>3</sub> and 0.3125 mLs HCl to each 50 mL vessel.

## 2. Solids

a. A 1.0  $\pm$ 0.02 gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and recorded in the sample digestion logbook.

## 3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

## B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

## VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 85° for oil samples. Record in digestion logbook.

## IX. PROCEDURE

### A. Glassware preparation for oil digestion or when the hot-block can not be used:

1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
2. Rinse glassware with reagent water that contains 5% HNO<sub>3</sub> and 5% HCl followed by a rinse with reagent water.
3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

### B. Aqueous sample filtration (for dissolved metals):

1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO<sub>3</sub> followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
2. Rinse a 0.45 micron filter with 1:5 HNO<sub>3</sub> thoroughly, followed by D.I. water.
3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
4. Discard the first 50 to 100 mL.
5. A preparation blank must be taken through the filtration step and analyzed with the sample.
6. Preserve the sample with HNO<sub>3</sub> to pH<2.
7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO<sub>3</sub>. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

### C. Aqueous sample preparation

1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".
  - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
  - b. Add 0.50 mL ( 1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample.
  - c. Add 2.5 mL ( 5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
  - d. Cover the sample with a ribbed watch glass or equivalent source.
  - e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, ( 12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
  - f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - g. Bring sample to its predigestion volume ( or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.
  - h. The sample is now ready for analysis.
  - i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
- 2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
  - b. Add 1.0 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Add 2.50 mL concentrated HCl to the sample.
  - d. Cover the sample with a ribbed watch glass or equivalent source.
  - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
  - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
  - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
  - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - i. The sample is now ready for analysis.
  - j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel.
  - b. Add 1.5 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Cover the sample with a ribbed watch glass.
  - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature

being recorded in the log book. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.

- e. Add another 1.5 mL portion of concentrated HNO<sub>3</sub> to the sample.
- f. Cover the sample with a ribbed watch glass.
- g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
- h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
- i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
- j. Cover the digestion vessel and reflux for an additional 15 minutes.
- k. Bring sample to its predigestion volume in digestion vessel.
- l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

**Note:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

- m. The sample is now ready for analysis.
  - n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
- b. Add 2.5 mL 1:1 HCl to the sample.
- c. Heat 15 minutes in a hot bath.
- d. Filter through a membrane filter.
- e. Adjust filtrate volume to 50 mL with DI water.
- f. Transfer to ICP analyst.

#### D. Solid sample preparation

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

#### **Grinding of Vegetation Samples**

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill

or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"

- a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
- b. Add 10 mL of 1:1 nitric acid ( $\text{HNO}_3$ ), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated  $\text{HNO}_3$ , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
- c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
- d. Continue to add 30%  $\text{H}_2\text{O}_2$  in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30%  $\text{H}_2\text{O}_2$ .)
- e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated  $\text{HNO}_3$ , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v)  $\text{HNO}_3$ . The sample is now ready for analysis.

- f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, “**Acid digestion of Sediments, Sludges and Soils**”

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

**NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.**

- c. Add 5 mL D.I. water and 5 mL concentrated  $\text{HNO}_3(1:1)$ , mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at  $95^\circ\text{C}$  for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book
- d. Allow the sample to cool. Add 5 mL concentrated  $\text{HNO}_3$ , replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by  $\text{HNO}_3$ , repeat this step (addition of 5 mL of concentrated  $\text{HNO}_3$ ) over and over until no brown fumes are given off by the sample indicating the complete reaction with  $\text{HNO}_3$ . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at  $95^\circ\text{C} \pm 5^\circ\text{C}$  for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of  $\text{H}_2\text{O}_2$  if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H<sub>2</sub>O<sub>2</sub> to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H<sub>2</sub>O<sub>2</sub>.)

- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
  - g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
  - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.
- NOTE1:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
- NOTE2:** To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.
- j. The sample is now ready for analysis.
  - k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## E. Oils

## 1. Method 3031, "Digestion Procedure for Oils"

**NOTE: THIS METHOD IS VERY TIME CONSUMING--  
DISCUSS SUB-CONTRACTING SAMPLES WITH  
YOUR SUPERVISOR AS SOON AS THEY COME IN  
THE DOOR.**

- a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.
  
- g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to potassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

- h. Cautiously add 1.0 mL concentrated  $H_2SO_4$ , and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The  $H_2SO_4$  can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

NOTE: To prevent a strong exothermic reaction,  $H_2SO_4$  should be added dropwise to all samples unfamiliar to the analyst and to all samples that are known to be highly reactive.

The reaction can take several seconds to begin, but when it occurs it will be very quick, vigorous, and exothermic. Generally larger sample sizes will react faster than smaller. Likewise, lower average molecular weight materials will react faster than heavier. Do not be misled by an initial lack of reactivity. A grey-white vapor will be ejected from the beaker ( $\text{SO}_3$ ) and splattering and bubbling can occur. The beaker will become very hot. This step is complete when no more gases are given off and the sample would be a thick black lumpy paste. Allow the beaker to cool as needed.

NOTE: Care must be taken when working with very light organic materials, such as diesel fuels, as they may flash. Generally, the lower the average molecular weight of the material correlates to a greater danger of flashing. The danger of flashing is reduced by adding the sulfuric acid dropwise.

NOTE: If more than 10% of the sample is aromatic material, such as xylene, only a little grey-white vapor will form. This will reduce accuracy and complicate nebulization. If there is a significant amount of non-hydrocarbon material, a sputtering reaction will occur and black  $\text{MnO}_2$  particulates will be given off. See section (b.) above under procedure.

- i. Add 2 mL of concentrated  $\text{HNO}_3$  and stir. This reaction will be slightly exothermic. If larger sample sizes are used, it is not always necessary to increase the volume of  $\text{HNO}_3$  proportionately, depending on analytical needs. Some reddish-brown vapor ( $\text{NO}_2$ ) may be given off. Allow the reaction to continue until complete, that is when the digestate no longer gives off fumes. Allow the beaker to cool as needed.
- j. Add 10 mL of concentrated  $\text{HCl}$  and stir. If larger sample sizes are used, it is not always necessary to increase the volume of  $\text{HCl}$  proportionately, depending on analytical needs. This reaction will be slightly exothermic and gas formation and foaming will occur. Lighter oils will foam more than will heavier oils. If excess foaming occurs, add water to prevent sample loss. Allow the beaker to cool as needed.
- k. Heat the beaker until there is no further gas evolution. (temperature should not exceed  $150\text{ }^\circ\text{C}$  to prevent volatilization). There may be additional foaming or other milder reactions which may result in overflow from the beaker. If excess foaming occurs, either remove the beaker from the heating source until foaming subsides or add

sufficient water to prevent overflow. The final digestate should be a clear yellow liquid with black or dark reddish-brown particulates.

- l. Filter the digestate through Whatman 41 filter paper and collect filtrate in a volumetric flask or beaker.
- m. Wash the digestion beaker and filter paper, while still in the funnel, with no more than 5 mL of hot HCl.

NOTE: The purpose of this next step is to recover antimony, barium, and silver that may not have been completely solubilized. If the sample is not being prepared for these analytes, the next step may be skipped.

- n. (Optional) After having washed the filter paper, remove the filter and residue from the funnel and place it back in the beaker. Add 5 mL of conc. HCl and place the beaker back on the heating source until the filter paper dissolves (temperature should not exceed  $150\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  to prevent volatilization). Remove the beaker from the heating source and wash the cover and sides with reagent grade water and then filter the residue and collect the filtrate in the same flask or beaker as in sections f. and g. above. Allow the filtrate to cool and quantitatively transfer to a volumetric flask. Bring to volume.
- o. (Optional) If the filtrate is collected in a beaker, the filtrate can be heated again to drive off excess HCl. This can reduce matrix effects in sample introduction (temperature should not exceed  $150\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  to prevent volatilization). When sufficient HCl has been removed, remove the beaker from the heating source, allow to cool, and then transfer the contents to a volumetric flask and bring to volume. However, if too much HCl is removed, barium, silver and antimony can be lost.
- p. Analyze the filtrate by ICP-AES. Depending on the final volume selected, the total solids in the digestate may be high enough to cause nebulization problems. Problems due to high dissolved solids may be corrected by 1) following optional Section i., 2) using internal standards, 3) using flow injection analysis, or 4) using other matrix correction procedures.

#### Manganese Removal Steps

NOTE: The purpose of these next steps is to remove the manganese in the digest by precipitating it as manganese ammonium phosphate

under alkaline conditions. Elements that do not form insoluble phosphates, such as arsenic, are filtered out and can be analyzed at lower concentrations.

- q. Take the digestate, or portion of digestate and reduce the volume to remove as much HCl as possible without going below 10 mL. Then add conc.  $\text{NH}_4\text{OH}$  until pH is 7 or greater. For most matrices, the digestate will change colors (often from yellow to brown) at pH 7. A mild exothermic reaction will occur immediately.
- r. Add at least 2 g ammonium phosphate for each 1 g of potassium permanganate used in the digestion and stir. An excess of phosphate is needed for good analyte recovery. Then add enough water and mix to ensure maximum precipitation. A pink or yellow silky amorphous precipitate, manganese ammonium phosphate, will form. If too much  $\text{NH}_4\text{OH}$  is used some of the manganese ammonium phosphate can be solubilized. Stir until precipitation is complete. Some ammonium phosphate may remain unreacted at the bottom of the beaker.
- s. Filter the digestate through Whatman 41 filter paper (or equivalent) and collect filtrate in a volumetric flask or beaker.
- t. Heat the filtrate to volatilize the ammonia (temperature should not exceed  $150\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$  to prevent volatilization). The volume of filtrate can be reduced by heating to no less than 10 mL. If too much water is removed as ammonium chloride formed will solidify. If this occurs, either add enough water to dissolve the solids or filter out the solids and wash the residue with deionized water. The filtrate can be analyzed by ICP-AES.
- u. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## X. CALCULATIONS

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

## XI. QUALITY CONTROL

- A. Digestion

1. Temperature blank
  - a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
  - b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
  
2. Blanks
  - a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples. **NOTE: The blank for OILs MUST include an analyte-free oil or explosive reactions can occur.**
  - b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
  - c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
  - d. Sample is given a unique identifier in the digestion log.
  
3. Laboratory Control Samples
  - a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
  - b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
  - c. For soil/sediment samples, a soil matrix standard reference material (SRM ) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
  - d. Sample is given a unique identifier in the digestion log.
  - e. Recoveries of standard reference materials or laboratory control samples spiked with organo-metallic standards recoveries should be **±25% of their true values for OILS.**

## 4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

**NOTE:** Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

## 5. Blank Spike

- a. This is required for certain projects.

## B. Sample Matrix

**NOTE:** Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

## 1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

**NOTE:** For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium  
Magnesium  
Sodium  
Potassium

- v. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)

**d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

## XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
  - 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
  - 1. Redigest another sample aliquot.
  - 2. Glass beaker dry for an extended period of time? Discard beaker.

## XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
  - 1. All metals glassware must be kept separate from all other laboratory glassware.
  - 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
  - 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
  
- F. Please consult Waste Disposal SOP-405, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### **Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT**

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

**DEFINITIONS** – Refer to SOP-431 for common environmental laboratory definitions.

**MERCURY ANALYSIS IN WATER**  
**BY MANUAL COLD VAPOR TECHNIQUE**  
**METHODS USEPA SW846 7470A and 245.1**  
**CLP-M 4.1 (NJDEP does not accept CLPILM 04.1**  
**after June, 2003), Addendum for USEPA CLP**  
**ILM 05.2**

**SOP NUMBER:** SOP-103

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**APPROVED BY:**   
**SECTION MANAGER**

  
**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** 01/28/09

**DATE OF LAST REVIEW** 01/28/09

## MERCURY ANALYSIS IN WATER BY MANUAL COLD VAPOR

### References:

SW846 Method 7470A  
USEPA Method 245.1  
USEPA SOW ILM04.1  
See Addendum for SOW ILM05.2

## I. SCOPE AND APPLICATION

- A. This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.
- B. In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.
- C. The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

## II. SUMMARY OF METHOD

- A. The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

### **III. SAMPLE HANDLING AND PRESERVATION**

- A. Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.
- B. The holding time for the mercury digestion is 28 days from time of sampling.

### **IV. INTERFERENCES**

- A. Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.
- B. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.
- C. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).
- D. Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

### **V. Safety**

- A. Normal accepted laboratory practices should be followed while performing this procedure.
- B. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

- C. Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.
- D. The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

## VI. EQUIPMENT/APPARATUS

- A. Perken Elmer Flow injection Mercury system
- B. Mod Block Digester set to maintain  $95\pm 2^{\circ}\text{C}$  for 2 hours.
- C. Polypropylene sample digestion vessels with snap or screw caps or equivalent.  
**Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.**

## VII. REAGENTS AND STANDARD PREPARATION

### A. REAGENTS

1. Concentrated sulfuric acid suitable for Hg determination.
2. Concentrated nitric acid suitable for Hg determination.
3. Stannous chloride: in a 1000 mL volumetric flask add approximately 500 mL D.I. water, 30 mL concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
4. 3% HCl Carrier Solution: Dilute 30 mL of concentrated metals grade HCl to one liter. Prepare fresh daily.
5. Sodium chloride-hydroxylamine chloride solution: dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.
5. Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mL of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring ( minimum of 3 hours if absolutely necessary ). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.

6. Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mL D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

## B. STANDARDS

### 1. Traceability

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

**NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes ( or calibrated Eppendorfs ). All standards, blanks, and samples are taken through the digestion process.

- a. Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.
- b. Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mL of stock solution to 1000 mL in a 1000 mL volumetric flask, with 1.5 mL concentrated HNO<sub>3</sub>.
- c. Calibration standards

- i. Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.
  - a. Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 30 mLs</u>
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

- iii. Appropriate reagents are added as below in the sample preparation section.
- iv. Prepare one vessel for each.
- v. It is necessary to digest the calibration standards.
- e. Calibration verification standards
  - i. Initial calibration verification ( ICV ) solution – 4.0 ug/L
    - a. Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)
    - b. Appropriate reagents are added as below in the sample preparation section.
    - c. It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.
  - ii. Continuing calibration verification ( CCV ) solution
    - a. Prepared from the primary source standard.

- b. Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.
  - c. Appropriate reagents are added as below in the sample preparation section.
  - d. It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.
- f. Digestion standards
- i. Laboratory control sample
    - a. Prepared from the secondary source standard.
    - b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.
    - c. Appropriate reagents are added as below in the sample preparation section.
    - d. This solution should be given a unique identifier in the digestion log.
  - ii. Matrix Spikes
    - a. Prepared from the secondary source working standard.
    - b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.
    - c. Appropriate reagents are added as below in the sample preparation section.

## VIII. CALIBRATION

A. Set up the instrument with proper operating parameters.

1. Perkin Elmer Flow Injection Mercury System (FIMS).

- i. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. **(Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)**
- ii. Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
- iii. Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
- iv. Turn on computer and go to icon "AA Win LAB Analyst".
- v. Go to method; select "Hg CAL 2" then OK.
- vi. Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.
- vii. Go to "Sample Info" and enter the order of the samples and other information that may be needed.
- viii. Save entered sample list under "Save ....sample info file" Note: description and batch ID are normally the date of analysis.
- ix. Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.
- x. Turn the printer on.
- xi. Connect all tubing to the pump and blocks.
- xii. Start the pump by going to "FIAS" and click the pump 1 Icon (120).
- xiii. The pump will start, then lock down and tighten the tubes onto the pump.
- xiv. Turn on the nitrogen tank, it should be above 500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.
- xv. The pressure gauge on the PE100 should be just below 100.
- xvi. Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock

them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

- xvii. Adjust the spring tension tubing until there is a constant “bubble of low rate” coming out to the waste tube.
- xviii. Place carrier tubes into carrier and stannous chloride tube into SnCl<sub>2</sub>. (click valve fill inject and make sure flow is correct and the line is rinsed).
- xix. Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.
- xx. Allow a few minutes for reagents to flow through the system before starting analysis.
- xxi. Calibrate: Go to “Auto” click on “Analyze”, click on “calibrate”.
- xxii. “Select Location” enter #'s to be ran, and then press “OK”. Samples are done in increments of 10 samples

B. Analyze the calibration standards as below.

- 1. New calibration points must be analyzed when the ICV analysis is not within  $\pm 5\%$ . **A curve must be analyzed daily for all projects especially USACE and CLP projects.**
- 2. The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of  $\geq 0.995$  ( USACE ) or 0.998 ( other ). If not, a new curve must be analyzed.

## IX. PROCEDURE

A. Glassware preparation

- 1. After use, samples are neutralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.
- 2. Acid clean the glassware used for mercury prep as follows:
  - a. Rinse with low Hg content 1:1 HCl.
  - b. Rinse with D.I. water.

- B. Label the vessels indicating which sample will be in each..
- C. Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the digestion log.
- D. Sample preparation

1. Transfer 30 mL, or an aliquot diluted to 30 mL of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

**NOTE:** Normally, an automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix get spiked. Check with your manager.

2. Add 1.5 mL of concentrated sulfuric acid to each vessel and mix.
3. Add 0.75 mL of concentrated nitric acid to each bottle and mix.
4. Add 4.5 mL potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mL). If the purple color does not persist after the addition of 7.5 mL  $\text{KMnO}_4$  the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

**NOTE:** The same amount of  $\text{KMnO}_4$  added to the samples should be present in the standards and blanks.

5. Add 2.4 mL of potassium persulfate to each vessel and mix. Cover.
6. Heat for 2 hours in the block digester at  $95 \pm 2^\circ\text{C}$  ( the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature ), cool.
6. Samples may be saved at this point if there is not time to run the whole set that day.

**NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.**

- E. Sample analysis

1. Set up the instrument as described in the calibration section above.
2. When ready to run samples, add 1.8 mL of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of  $\pm 10\%$  for SW846-7470 and  $\pm 5\%$  for 245.1. Followed by the ICB ( $< \pm MDL$  for USACE or  $\pm RL/CRDL$  for others and CLP).
3. Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of  $\pm 20\%$  for SW846-7470 and  $\pm 10\%$  for 245.1
4. CCB must always follow the CCV. Control limits are ( $< \pm MDL$  for USACE or  $\pm RL/CRDL$  for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected  $> 2xMDL$  for DOD QSM Ver. 3.**
5. The autosampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	ICV
10	ICB
11	LCSW
AS LOC	Sample ID
12	PBW
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	CCV

22	CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	FCV
34	FCB

F. Data reporting

1. Reduce data to result which will be reported.
2. Complete the data review checklist ( attached ). Must be completed and attached to each set of USACE data.

**X. CALCULATIONS**

- A. Apply a least squares fit to the calibration standards plotting  $\mu\text{g Hg/L}$  versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume ( the 0.1  $\mu\text{g Hg}$  standard will be input as 1.0  $\mu\text{g Hg/L}$  ) ( 0.1 $\mu\text{g Hg}$  / 0.030 L solution ).
- B. Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.
- C. Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.
- D. Report the data as  $\mu\text{g Hg/L}$  of sample.

**XI. QUALITY CONTROL (Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)**

A. Daily

1. **The instrument must be calibrated daily for all projects.**

2. Begin each analysis with an ICV(QCS) second source. The control limits are  $\pm 10\%$  and IPC(CCV) for 245.1, limits are  $\pm 5\%$  and subsequent analyses are  $\pm 10\%$ .
  3. Analyze ICB. Control limits ( $< \pm \text{MDL}$  for USACE or  $\pm \text{RL/CRDL}$  for others and CLP), depending on method. **No analyte detected  $> 2 \times \text{MDL}$  for DOD QSM Ver. 3.**
  4. If the ICV(QCS) is not in control a new curve must be analyzed prior to sample analysis.
  5. If the IPC(initial CCV) for 245.1 is not within the limits of  $\pm 5\%$ , try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.
  6. Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are  $\pm 20\%$  for SW846-7470 and  $\pm 10\%$  for 245.1.
  7. A CCB must always follow a CCV, the control limit is ( $< \pm \text{MDL}$  for USACE or  $\pm \text{RL/CRDL}$  for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected  $> 2 \times \text{MDL}$  for DOD QSM Ver. 3.**
- B. Quarterly or as needed when doing straight CLP work.
1. IDL's for CLP 4.1.
- C. Digestion
1. LCS data should be maintained and available for easy reference or inspection.
  2. Preparation blank ( $< 1/2 \pm \text{RL}$  or  $\pm \text{RL/CRDL}$  for common contaminants (DOD) and  $\pm \text{RL/CRDL}$  for others and CLP).
    - a. Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a

sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.

3. Laboratory control sample ( LCS )
  - a. Employ a minimum of one laboratory control sample ( LCS ) per sample batch to verify the digestion procedure. The LCS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are  $\pm 20\%$  for SW846-7470 and  $\pm 15\%$  for 245.1. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified. The project manager or QA Officer will make this determination.

#### D. Sample matrix

1. Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. CLP does not allow this. Project specific requirements will take precedence in these situations.
2. Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within  $\pm 25\%$  of the true value ( **$\pm 20\%$  for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.

**NOTE:** For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control

limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.

4. For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be  $\pm 10\%$ . The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.
  5. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.
- E. Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

**TABLE I**

<b>Aqueous Method Detection Limits(MDL), Empirical Laboratories' Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>				
<b>Mercury by EPA 245.1, 7470A, SOW 4.1 &amp; 5.2</b>	<b>AQUEOUS MDL(ug/L)</b>	<b>AQUEOUS ERL(ug/L)</b>	<b>AQUEOUS CRQL ILMO 4.1 (ug/L)</b>	<b>AQUEOUS CRQL ILMO 5.2 (ug/L)</b>
<b>Mercury</b>	0.08	0.20	0.2	0.2

**TABLE 2**

<b>ANALYTE</b>	<b>WAVELENGTH</b>
<b>Mercury</b>	<b>253.7</b>

## XII. CORRECTIVE ACTIONS

### A. INSTRUMENT RELATED

1. ICV(QCS for 245.1)- second source not within  $\pm 10\%$ .
  - a. If the problem is with the solution.
    - i. Reprepate, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within  $\pm 20\%$  for SW846 and  $\pm 10\%$  for (245.1,  $\pm 5\%$  for initial IPC and  $+ 10\%$  for subsequent IPCs)
  - a. If the problem is with the solution.
    - i. Reprepate, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and reprepate/reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalyzed.

### B. DIGESTION RELATED

1. The preparation blank less than  $<1/2$  RL or  $\pm RL/CRDL$  for common contaminants (DOD) and  $\pm RL/CRDL$  for others and CLP.
  - a. If the problem is with the instrument or stannous chloride.
    - i. Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepate the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.
  - b. If the problem is with the digestion.
    - i. All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation

blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits ( or  $\pm 20\%$ ,  $\pm 15\%$  for **245.1** ).
  - a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control if further sample bottles are available.
  - b. Is the problem is with the digestion.
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

### C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$  ( **$\pm 20\%$  for DOD projects**)
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.
3. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

### XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### XIV. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 7470A*
2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B*
3. *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

## **XV. DEFINITIONS**

1. Refer to SOP-431 for common definitions.

### **ADDENDUM FOR USEPA SOW ILM05.2**

1. The CCV concentration must be different from the ICV.
2. The same CCV shall be used throughout analysis for an SDG.
3. Calibration standards must be within 5% of the standard concentration.
4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is  $\pm 30\%$ .
5. Spike samples at 1 ug/L for water.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method:</b> 7470A ( Mercury )

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____

15. "Special" sample preparation and analytical requirements have been met. \_\_\_\_\_
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete). \_\_\_\_\_

Comments on any "No" response:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**MERCURY ANALYSIS IN SOIL/SEDIMENT**  
**BY MANUAL COLD VAPOR TECHNIQUE**  
**METHODS SW846 7471A 7471B, EPA 245.5 and**  
**CLPILM 04.1 (NJDEP does not accept CLPILM**  
**04.1 after June, 2003), Addendum for USEPA CLP**  
**ILM 05.2**

SOP NUMBER:

SOP-104

REVISION NUMBER:

17

APPROVED BY:

*Betty DeVill*

SECTION MANAGER

*Randy P. Ward*

TECHNICAL DIRECTOR

EFFECTIVE DATE:

01/29/09

DATE OF LAST REVIEW

01/29/09

**MERCURY ANALYSIS IN SOIL/SEDIMENT BY  
MANUAL COLD VAPOR TECHNIQUE**

**References:**

**SW846 Method 7471A, 7471B  
USEPA Method 245.5, CLP SOW ILM04.1  
See Addendum for CLP SOW ILM05.2**

**I. SCOPE AND APPLICATION:**

- A This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.
- B. The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

**II. SUMMARY OF METHOD:**

- A. A weighed portion of the sample is acid digested for 2 minutes at 95±2°C, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

**III. SAMPLE HANDLING AND PRESERVATION:**

- A. Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.
- B. Refrigerate solid samples at 4°C (±2°C) upon receipt until digestion and analysis.
- C. The sample should be analyzed without drying. A separate percent solids determination is required
- D. The holding time for digestion of mercury samples is 28 days.

#### IV. INTERFERENCES:

- A. Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.
- B. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.
- C. Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.**
- D. 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 is present.

#### V. SAFETY

- A. Normal accepted laboratory practices should be followed while performing this procedure.
- B. The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- C. Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

**VI. EQUIPMENT/APPARATUS:**

- A. Perkin Elmer Flow Injection Mercury System (FIMS)
- B. Perkin Elmer AS 90
- C. Mercury lamp
- D. Environmental Express Mod-Block digestion block capable of holding  $95 \pm 2^{\circ}\text{C}$  for 2 hours
- E. A scale or balance capable of weighing to  $0.01 \pm 0.02$  gram.
- F. Snap cap digestion polypropylene vessels for use with the mod block digester.  
**Five vessels of each lot must be taken through analysis to check for mercury**
- G. Polypropylene watch glasses suitable for use with the above vessels in F above.
- H. Manual Sample Mill
- I. Wiley Sample Mill
- J. Clippers for cutting vegetation

**VII. REAGENTS AND STANDARD PREPARATION:****A. REAGENTS**

1. Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.
2. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO<sub>3</sub>. Both HNO<sub>3</sub> and HCl must be of the reagent grade suitable for mercury determinations.

**NOTE:** This reagent is required for use when USACE project samples are being digested.

3. Concentrated HCl.
4. Concentrated HNO<sub>3</sub>.

5. Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H<sub>2</sub>O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.
6. Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note : this is normally made up 2 liters at a time.
7. Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.
8. 3 % HCl carrier solution: 30 mL HCl – 1 L DI H<sub>2</sub>O; Prepare fresh daily.
9. Potassium persulfate 5% solution: Dissolve 100g in 2 liters of D.I. water. Used with digestion of CLP soils.

## B. STANDARDS

### 1. Traceability

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

### 2. Preparation

**NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes ( or calibrated Eppendorfs ). All Standards, blanks, and samples are taken through the digestion process.

- a. Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.
- b. Primary source and secondary source mercury standard solutions: dilute 2 mL of stock solution to 1000 mL in a 1000 mL volumetric flask, with 1.5 mL concentrated HNO<sub>3</sub> (200 ug/L).
- c. Calibration standards
  - i. Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.
    - a. Dilute the volumes below to 5 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 5 mLs for the initial step of the digestion.)

<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 50 mL</u>
0.20	0.050
0.50	0.125
1.0	0.25
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

- ii. Appropriate reagents are added as below in the sample preparation section.
  - iii. Prepare one vessel of each.
  - iv. It is necessary to digest the calibration standards when following all mercury methods.
- e. Calibration verification standards
  - i. Initial calibration verification ( ICV ) solution – 4.0 ug/L
    - a. Prepared from the secondary source mercury standard (200 ug/L).
    - b. Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.
    - c. Appropriate reagents are added as below in the sample preparation section.

- d. It is necessary to digest the ICV standards when using all mercury methods for soil.
  
- ii. Continuing calibration verification (CCV) solution
  - a. Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.
  - b. Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mL with reagent water in a polypropylene digestion vessel.
  - c. Appropriate reagents are added as below in the sample preparation section.
  - d. It is necessary to digest the CCV standards when following all mercury methods for soil.
  
- f. Digestion standards
  - 1. Laboratory control sample
    - a. The Laboratory Control Sample (LCS) is prepared from the secondary source mercury standard (200 ug/L) and added to ~ 0.3 grams of teflon chips.
    - b. Prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of teflon chips.
    - c. Appropriate reagents are added as below in the sample preparation section.
    - d. This solution is given a unique identifier in the digestion log.
  
  - 2. Matrix Spikes
    - a. Prepared from the primary or secondary source mercury standard (200 ug/L).
    - b. Prepared by adding 0.50 mL of the mercury standard (200 ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.
    - c. Appropriate reagents are added as below in the sample preparation section.

## VIII. CALIBRATION:

### A. Set up the instrument with proper operating parameters.

#### 1. Perkin Elmer Flow Injection Mercury System (FIMS)

- a. Prepare the instrument for calibration by the following steps:
  - i. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. (**Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.**)
  - ii. Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
  - iii. Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
  - iv. Turn on computer and go to icon "AA Win LAB Analyst"
  - v. Go to method; select "Hg CAL 2" then OK.
  - vi. Wavelength = 253.7; smoothing points =9; measurement = peak height; read time = 18 sec.; BCC time = 2 sec.
  - vii. Go to "Sample Info" and enter the order of the samples and other information that may be needed.
  - viii. Save entered sample list under "Save ...sample info file"  
Note: description and batch ID are normally the date of analysis.
  - ix. Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information." File. The other is to select a results library. Double click on heading and choose.
  - x. Turn the printer on.
  - xi. Connect all tubing to the pump and blocks.
  - xii. Start the pump by going to "FIAS" and click the pump 1 Icon (120).
  - xiii. The pump will start, then lock down and tighten the tubes onto the pump.
  - xiv. Turn on the nitrogen tank, it should be >500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.
  - xv. The pressure gauge on the PE100 should be just below 100.
  - xvi. Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.
  - xvii. Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

- xviii. Place carrier tubes into carrier and stannous chloride tube into SnCl<sub>2</sub>. ( click valve fill inject and make sure flow is correct and the line is rinsed)
- xix. Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.
- xx. Allow a few minutes for reagents to flow through the system before starting analysis.
- xxi. Calibrate: Go to “Auto” click on “Analyze”, click on “calibrate”.
- xxii. “Select location” enter the #'s of the samples to be analyzed, then “OK”.

B. Analyze the calibration standards as below.

- 1. A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within  $\pm 10\%$  for SW846 7471A and 245.5 methods, or  $\pm 20\%$  for 7471B.
- 2. The curve should be linear with a calculated intercept with a minimum correlation coefficient(r) of  $\geq 0.995$  (USACE ) or 0.998 ( other ). If not, a new curve must be analyzed.
- 3. **CLP requires a blank + 5 calibration standards (0, .02, .05, .1, .5 and 1.0  $\mu\text{g}$ ). (One standard must be at CRDL or IDL whichever is greater.)**

**IX. PROCEDURE:**

A. Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.

B. Sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
- Two quarters should then be mixed to form halves.
- The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed.

**NOTE: Samples that are clay type materials must be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

### **Grinding of Vegetation Samples**

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. Transfer 0.30 g ( for USACE work use anywhere from 0.20 to 1.0 g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and in the digestion log. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. Record in specific balance calibration log.)
2. Add 2.5 mL of reagent water, and 2.5 mL of aqua regia and mix for samples. Add 2.5 mL of aqua regia to standards and mix.
3. Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at  $95 \pm 2^\circ\text{C}$  (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature ).
4. Cool, bring to 30 ml with D.I. water
5. Add 7.5 mL potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mL).

**NOTE:** The same amount of  $\text{KMnO}_4$  added to the samples should be present in the standards and blanks.

6. Heat for 30 minutes on the hot block at  $95 \pm 2^\circ\text{C}$  ( The temperature must be monitored and documented. Record observed temperature, correction

factor, and the corrected temperature ), cool. Samples may be saved at this point if there is not time to run the whole set that day.

7. Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.
8. Bring to 50 ml with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. The samples are now ready for analysis.

**NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.**

### C. Sample analysis

1. Set up the instrument as described in the calibration section above.
2. When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step "3". **NOTE:** When aqua-regia is added assume that all samples and standards have chlorine and treat accordingly. Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.
3. Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits of  $\pm 10\%$  for SW846-7471A or  $\pm 20\%$  for 7471B and  $\pm 5\%$  for 245.5 methods.
4. The ICB must follow the calibration standards ( $< \pm \text{MDL}$  (USACE) or  $\pm \text{RL}/\text{CRDL}$  for other or CLP), but not before the ICV. **No analyte must be detected  $> 2 \times \text{MDL}$  for DOD QSM Ver. 3.**
5. Each set of ten samples must be followed by a CCV with control limits of  $\pm 20\%$  for SW846-7471A and B and  $\pm 10\%$  for 245.5 method. The run must also end with a CCV, then CCB.
6. Analyze CCB after calibration and each CCV. The CCB frequency is 10% or every 2 hours whichever is more frequent. (control limit is  $< \pm \text{MDL}$  or  $\pm \text{RL}/\text{CRDL}$  for other or CLP). **For DOD QSM Ver.3 CCB at**

**beginning and end of sequence and after every 10 samples. No analyte detected > 2xMDL.**

7. Instrument Run Log example:

<b>AS LOC</b>	<b>Sample ID</b>
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
<b>AS LOC</b>	<b>Sample ID</b>
5	0.2
6	0.4
7	0.6
8	1.0
9	ICV
10	ICB
11	LCSW
12	PBW
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	CCV
22	CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	FCV
34	FCB

8. Sample analysis:

- i. Go to “Analyze”, “select location” and type in the range of numbers needed to complete analysis. (ie. 9-54). Press enter and the autosampler will proceed to enter the selected range. NOTE: Check standards are loaded as part of the tray.
- ii. Make sure that the sample wash beaker is filled with 3% HCl.
- iii. Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL are reported flagged as estimated, (“B” flag).

D. Data reporting

1. Reduce data to result which will be reported using the soil spreadsheet found on the network..
2. Complete the data review checklist ( attached ). Must be completed and attached to each set of USACE data.

**X. CALCULATIONS:**

- A. Pull up the blank spreadsheet at K:\wcm\tests\mercury and fill in all the information pertinent to the current analysis. Save as the date of analysis. This information can be obtained from your mercury digestion log.
- B. Input the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.
- C. Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.
- D. The spreadsheet should divide the result which is the  $\mu\text{g Hg}$  obtained from the sample mass by the sample mass in grams. This will yield a result of  $\mu\text{g Hg/g}$  sample on a wet weight basis. Calculations in the spreadsheet should be checked occasionally to make sure that they are working correctly.
- E. If available, divide the result by the %solids to obtain the result on a dry weight basis.
- F. Report the data as  $\mu\text{g Hg/g}$  of sample (mg/kg wet or mg/kg dry when % solids are available).

**XI. QUALITY CONTROL (Reference SW-846, 7471A Update III, 7471B Revision 2 February 2007, USEPA CLP ILMO 4.1 or EPA 245.5 for further clarification)**

A. Daily

1. **The instrument must be calibrated daily for all projects.**
2. Begin each analysis with an ICB. The control limits are  $\pm 10\%$  for 7471A and 245.5,  $\pm 20\%$  for 7471B.
3. Analyze ICB. Control limit is  $< \pm \text{MDL}$  or  $\pm \text{RL}/\text{CRDL}$  for other or CLP. **For DOD QSM Ver. 3, no analyte detected  $> 2x \text{MDL}$ .**
4. If the ICB is not in control a new curve must be analyzed prior to sample analysis.
5. Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. The control limits are  $\pm 20\%$  for SW846-7471A, SW846 7471B and for 245.5. If an exceedance occurs, analyze another CCV, if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.
6. Follow each CCV with a CCB. Control limit is  $< \pm \text{MDL}$  or  $\pm \text{RL}/\text{CRDL}$  for others or CLP. **For DOD QSM Ver. 3, no analyte detected  $> 2x \text{MDL}$ .**

B. Quarterly

1. IDLs for CLP (Follow SOP - 414).

C. Annually

1. MDLs must be analyzed for all matrixes (Follow SOP - 414).

D. Digestion

1. LCS data should be maintained and available for easy reference or inspection.
2. Preparation blank ( $< \pm 1/2 \text{RL}$  or  $\pm \text{RL}$  for common contaminants or  $\pm \text{RL}/\text{CRDL}$  for others or CLP)
  - a. Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be  $< \pm 1/2 \text{RL}$  for USACE or  $\pm \text{RL}/\text{CRDL}$  for others or CLP. If not, the analyst must use good judgment to evaluate the impact upon

the associated samples. There is no impact if an associated sample is below the method detection limit or if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit, but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA officer will make this determination.

3. Laboratory control sample ( LCS ).

- a. Employ a minimum of one LCS per sample batch to verify the digestion procedure.. The LCS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are  $\pm 20\%$  for SW846-7471A, 7471B and 245.5 solid samples. An LCS will accompany each batch of soil samples. If the LCS is not in control, the Inorganic Manager and QA Officer must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This corrective action is documented in a CAR.

E. Sample matrix

1. Analyze one replicate sample for every twenty samples 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. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations.
2. Analyze one spiked sample and spiked sample duplicate for every twenty samples 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. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within  $\pm 25\%$  for 7471A and  $\pm 20\%$  for 7471B of the true value(  **$\pm 20\%$  for DOD projects**). If results do not fall within the control limit-redigestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your supervisor. A Corrective Action Report (CAR) must be filled out and attached to the

data as well as emailed or sent to the supervisor when the control limits are exceeded.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.
  
4. For 245.5 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be 10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.
  
5. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

**F. Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:**

**TABLE I**

<b>Solid/Soil Method Detection Limits(MDL), Empirical Laboratories' Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>				
<b>Mercury by EPA 245.1, 245.5, 7471A, SOW 4.1 &amp; 5.2</b>	<b>SOLID/SOIL MDL (mg/Kg)</b>	<b>SOLID/SOIL ERL (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)</b>
<b>Mercury</b>	0.0157	0.033	0.1	0.1

**TABLE 2**

ANALYTE	WAVELENGTH
Mercury	253.7

**XII. CORRECTIVE ACTIONS**

**A. INSTRUMENT RELATED**

1. ICV not within  $\pm 10\%$  (SW846) and (245.5)
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and recheck ICV.
  
2. CCV not within  $\pm 20\%$  (SW846) and (245.5)
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalysed.

**B. DIGESTION RELATED**

1. The preparation blank less than  $\pm \frac{1}{2}$  RL for DOD or  $\pm$ RL/CRDL for others or CLP.
  - a. If the problem is with the instrument or stannous chloride.
    - i. Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.
    - ii. If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.
  - b. If the problem is with the digestion.
    - i. All associated samples which are below the method detection limit (MDL) or have a level of mercury greater than 10X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits.
  - a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control with another aliquot of the sample.
  - b. If the problem is with the digestion.
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

### C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$  7471A and  $\pm 20\%$  7471B ( **$\pm 20\%$  for DOD projects**)
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

### XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### XIV. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III/IV); Method 7471A, 7471B*
2. *USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 245.1; APX-B*
3. *USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

### XV. DEFINITIONS

1. Refer to SOP-431 for common definitions.

**Addendum for USEPA CLP ILM 05.2**

1. CCV concentration must be different from ICV.
2. The same CCV shall be used throughout analysis for a sample delivery group.
3. Calibration standards must be within 5% of the standard concentration.
4. 0.2 grams of sample must be used for the sample aliquot, add enough reagent water to each sample to make a total volume of 10 mL. Proceed with method as in the water method SOP 103.0 Revision 9.
5. The ICV and CCV must be at  $\pm 20\%$  recovery.
6. A CRA must be analyzed at the beginning and end of each batch of 20 samples. Right after the ICV/ICB and right before the final CCV/CCB. The control limit is  $\pm 30\%$ .
7. The matrix spike must be analyzed at the concentration of 0.5 mg/Kg.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method:</b> 7471A (Mercury)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

**ANALYST DATA REVIEW CHECKLIST  
7471A (Mercury)**

Comments on any "No" response:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**METALS ANALYSIS**

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**BY INDUCTIVELY COUPLED PLASMA-  
ATOMIC EMISSION SPECTROMETRY (ICP-  
AES) TECHNIQUE**

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**METHODS 200.7, ( SW846) 6010B, (SW846)  
6010C, (SM 19<sup>th</sup> Edition 2340B) Hardness  
Calculation, (USEPA CLP) ILMO 4.1 (NJDEP  
does not accept CLPILM 04.1 after June, 2003)  
Addendum for USEPA CLPILM 05.2**

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**SOP NUMBER:**

**SOP-105**

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**REVISION NUMBER:**

**15**

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**APPROVED BY:**

*Betty DeVillb*

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**SECTION MANAGER**

*Randy D. Ward*

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**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:**

**02/22/09**

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**DATE OF LAST REVIEW:**

**05/08/09**

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## ICP METHOD SOP

**References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19<sup>th</sup> Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2**

### I. SCOPE AND APPLICATION

- A. Inductively Coupled Argon Plasma (ICAP) determines trace elements in solution. **We use the ICP to determine the concentration of the following metals: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V and Zn.** All matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.
- B. **Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.** Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

### II. SUMMARY OF METHOD

- A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

- B. This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the the original sample. 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 analytic wavelength measured.

Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based iTEVA software.

- C. ICP's primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.
- D. It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium internal standard cannot be used.)

### III. SAMPLE HANDLING AND PRESERVATION

- A. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).
- B. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. Since the most concentrated acid matrix allowed for direct disposal down an acid sink is a ratio of 20:1, the samples must be diluted with 1 part water to 2 parts sample prior to pouring down the sink while the tap water is running.
- C. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.
- Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO<sub>3</sub> prior to metal determination.

- Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.
- Standard Methods 19<sup>th</sup> Edition Method 3030C prepares groundwaters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO<sub>3</sub>, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.
- Method 3050B prepares waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

#### IV. INTERFERENCES

- A. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.
3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
4. When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2 from the method, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments

may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

5. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.
7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
8. When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples

analyzed is such they do not contain concentrations of the interfering elements at  $\pm$  one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

- B. 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. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.
- C. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.
- D. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration

accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

## **V. SAFETY**

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
  - 1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
  - 2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
  - 3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

## **VI. EQUIPMENT/APPARATUS**

- A. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- B. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- C. Radio frequency generator compliant with FCC regulations: Thermo Scientific or equivalent.
- D. Argon gas supply – Liquid Argon
- E. Class A volumetric flasks
- F. Class A volumetric pipettes
- G. Analytical balance - capable of accurate measurement to a minimum of three significant figures (.001gm): Mettler model AE100
- H. Variable Eppendorf Pipettes 1000 $\mu$ L; 5000 $\mu$ L

## **VII. REAGENTS AND STANDARD PREPARATION**

### **A. Notes**

1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
2. 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. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

## **B. REAGENTS**

1. Hydrochloric acid (concentrated), HCl.
2. Nitric acid (concentrated), HNO<sub>3</sub>.

## **C. STANDARDS**

### **1. Matrix**

- a. All standards contain 2% HNO<sub>3</sub> and 5% HCl.

### **2. Storage**

- a. The standards are stored at room temperature in 500 mL Teflon bottles.

### **3. Traceability**

- a. All records shall be maintained on all reference materials within Element. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique Element number that is recorded on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date in Element. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations,

volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

#### 4. Calibration standards

- a. All standards have an acid matrix of 2% HNO<sub>3</sub> and 5% HCl and should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs).
- b. STD-1 is the calibration blank: Reagent grade water **matrix matched as in (a) above. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**
- c. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.
- e. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.
- f. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- g. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- h. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- i. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- j. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

- k Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- l Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- m Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- n Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- o Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- p Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- q Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- r Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- s Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- t Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- u Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- v Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

- w. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- x. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- y. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- z. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- aa. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- bb. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- cc. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- dd. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- ee. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- ff. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- gg. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

## 5. Calibration and Calibration Verification standards

- a. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.
- b. The CRI solution is analyzed to check the accuracy of the instrument down near the contract required detection limits (CRDL). It is analyzed in conjunction with the interference check sample. The sample is prepared from a purchased solution which contains 120 µg/mL Sb, 100 µg/mL Co and V, 80 µg/mL Ni, 50 µg/mL Cu, 40 µg/mL Zn, 30 µg/mL Mn, 20 µg/mL As, Cr, Ag and Tl, 10 µg/mL Be, Cd and Se along with 6 µg/mL Pb. 500 µL of the solution is diluted to 500 mL. This solution is stable for 6 months.
- hh. The interference check solutions ( ICSA and ICSAB ) are prepared to contain known concentrations of interfering elements that will provide an adequate test of the IECs. A solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the ICSA. The ICSAB is prepared by diluting 100x a solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly.
- d. Reporting Limit Standard- Prepared 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B diluted to 100 ml with 2% HNO<sub>3</sub> and 5% HCL matrix , mix well. Solution stable for 3 months

## 6. Digestion standards

- a. The Laboratory control sample ( LCS ) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3. 25 mL of HCl and 10 mL of HNO<sub>3</sub> are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the LCS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier. The LCS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. Note: The analysis of Molybdenum is not a routine procedure but a project-specific requirement. A customized LCSW mix must be prepared to contain this target analyte.
- b. The solid Laboratory Control Sample (Soil) (LCSS) is prepared by weighing up 1.0 g of teflon chips and spiking using the same spiking

solutions used to spike the sample matrix. This standard is given a unique identifier i.e. LCSS(date prepared)A,B,C etc.

- c. The spiking solutions are prepared as follows:
1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier. Add 0.050 mL (0.20 mL for soil samples) of CLP-CAL-1 solutions A and B, and 0.0125 mL (0.05 mL for soil samples) of CLP-CAL-2 and 3 to 50 mL of sample (1gram of sample for soils) for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.
  2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:  
2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.
  3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:  
0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

## VIII. CALIBRATION AND ASSOCIATED QA/QC

- A. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- B. Operating conditions - **The instrument settings can be found in method file.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are 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 individual analyte line on that particular instrument. 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.
- C. Autopeak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. (**See SOP-106, ICP Instrument Operation**) Flush the system with 2% HNO<sub>3</sub> / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ( $r \geq 0.998$ ). If a three point calibration curve is not required for the client samples being analyzed Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.
- D. Before beginning the sample run, analyze the Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ( $\pm 10\%$ ) for 200.7 ( $\pm 5\%$ ) and ICB ( $< \pm MDL$  or  $\pm RL/CRDL$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $> 2xMDL$** ) first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within  $\pm 20\%$  for DOD projects and  $\pm 30\%$  for samples analyzed for 6010C. Then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition.
- E. For CLP projects, verify the validity of the curve in the region of 2x the contract required detection limit ( CRDL ) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB

criteria:  $< \pm\text{MDL}$  or  $\pm\text{RL/CRDL}$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $>2x\text{MDL}$ , beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Results should be within  $\pm 20\%$ . Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

- F. Verify the interelement and background correction factors at the beginning and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB criteria:  $< \pm\text{MDL}$  or  $\pm\text{RL/CRDL}$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $>2x\text{MDL}$ , beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check solution A and AB. Results should be within  $\pm 20\%$  of the true value for ICSAB. **For ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes  $< 2x\text{MDL}$ .**(CRI, ICSA and ICSAB required at the end for CLP projects only).

- G. *When analyzing samples associated with North Carolina or with DOD QSM Ver. 3 work, a solution containing analytes at their reporting limit must be analyzed prior to sample analysis. The concentrations must be within 20% DOD( 20 or 30% depending on project) of their true values to be acceptable.*

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

- H. The instrument must be calibrated once every 24 hours if performing straight CLP work.
- I. Instrument Autosampler Report example:

#### **Calibration Rack(used by instrument software to insert QC)**

- 1) STD 1-blank
- 2) Low Cal
- 3) Mid Cal
- 4) Ba @ 5000 ppb
- 5) QC5
- 6) QC 21
- 7) NAK 100
- 8) QC3

#### **Sample Sequence RACK 1**

- 1) Al IEC-(readback)

- 2) Fe IEC-(readback)
- 3) ICV
- 4) ICB-initial
- 5) RL-reporting limit standard
- 6) Ba@ 5000 ppb (readback)
- 7) QC5
- 8) NAK High-(readback)
- 9) QC 21 High-(readback)
- 10) Salt Cal at 500 ppm (readback)
- 11) Rinse
- 12) CRI-0
- 13) ICAS-0
- 14) ICASB-0
- 15) Rinse
- 16) CCV 1A
- 17) CCB 1A
- 18) Preparation Blank (*Batch #* BLK-1)
- 19) Laboratory Control Sample (*Batch #* BS-1)
- 20) Sample 1
- 21) Sample 2
- 22) Sample 3
- 23) Sample 4
- 24) Sample 5
- 25) Sample 6
- 26) Sample 7
- 27) Sample 8
- 28) CCV 1B
- 29) CCB 1B
- 30) Sample 9
- 31) Sample 10
- 32) Sample 11
- 33) Sample 12
- 34) Sample 13
- 35) Sample 14
- 36) Sample 15
- 37) Sample 16
- 38) Sample 17
- 39) Sample 18
- 40) CCV 2A
- 41) CCB 2A
- 42) Sample 19
- 43) Sample 20
- 44) Sample matrix spike (*batch#* MS-1)
- 45) Sample matrix spike duplicate (*batch#* MSD-1)
- 46) Sample post digestion spike (*batch#* PS-1)
- 47) Sample serial dilution (*batch#* SRD-1)
- 48) CRI-1

- 49) ICSA-1
- 50) ICSAB-1
- 51) Rinse
- 52) CCV 2B
- 53) CCB 2B
- 54) Preparation Blank (*batch#* BLK-1)
- 55) Laboratory Control Sample (*batch#* BS-1)
- 56) Sample 1
- 57) Sample 2
- 58) Sample 3
- 59) Sample 4
- 60) Sample 5

## **RACK 2**

- 1) Sample 6
- 2) Sample 7
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

## **IX. PROCEDURE**

- A. Once the instrument has been calibrated, begin the analysis of samples.
- B. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**
- C. Flush the system with 2% HNO<sub>3</sub> / 5% HCl for at least 1 minute before the analysis of each sample.
- D. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7,  $\pm 10\%$  of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and RL are flagged as estimated with a "B" flag.**
- E. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

- The results of the CCV are to agree within 10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from replicate ( minimum of two integrations ). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.
- The results of the calibration blank (this is not the method/preparation blank) are to agree within <math>\pm\text{MDL}</math>(SW-846 Method 6010B), and 3 x IDL or CRDL for CLP, for **DOD QSM Ver. 3 no analytes detected >2xMDL**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).
- Total hardness is reported from HNO<sub>3</sub> preserved sample. The final concentration is calculated from the calcium and magnesium results as follows:

$$\text{Ca mg/L} \times 2.5 + \text{Mg mg/L} \times 4.1 = \text{total Hardness in mg/L as CaCO}_3$$

- F. Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## X. CALCULATIONS

- A. The instrument will generate data results in mg/L or µg/L ( labeled appropriately). Each result represents an average of three individual readings per metal channel.
- B. For aqueous samples, if a post/predigestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.
- C. For solid samples, if a postdigestion dilution is performed , the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ ( ug/g or mg/kg )} = IR * DF * FED / SM$$

SR	=	Sample result
IR	=	Instrument result ( $\mu\text{g/L}$ )
DF	=	Dilution factor ( post digestion )
FED	=	Final volume of digestate ( L )
SM	=	Sample mass digested( g )

## **XI. QUALITY CONTROL**

### **A. Daily**

1. See sections VIII and IX above.

### **B. Quarterly**

1. Linear range standards must be analyzed at a frequency no less than once every three months. The linear range standard represents the second standard required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-5% of true value.
2. The interelement correction factors ( IEC ) should be verified at the time the linear range standards are analyzed.
3. IDL's if CLP work required.

### **C. Digestion**

1. All quality control data should be maintained and available for easy reference or inspection.
2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank, sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than  $\pm \frac{1}{2}$  RL (USACE) or  $\pm RL/CRDL$  for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified.
3. Employ a minimum of one laboratory control sample ( LCS ) for aqueous samples or one teflon chip spiked sample per sample batch to verify the digestion procedure. These LCSs are taken through the same digestion/preparation steps as the sample being tested. The control limits

are  $\pm 15\%$  method 200.7 - aqueous and soil samples or  $\pm 20\%$  for all other methods aqueous and soil samples. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be redigested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

#### D. Sample

1. Analyze one replicate sample for every twenty samples 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. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are 20% RPD (if both are  $>5x$  CRDL) or  $\pm$  the CRDL ( if either are  $<5X$  CRDL).
2. Analyze one spiked sample and spiked sample duplicate for every twenty samples 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. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within  $\pm 25\%$  of the true value ( **$\pm 20\%$  for DOD projects**). If not, a post digestion spike should be analyzed.
3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than five times the contract required detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.
4. *The following should be analyzed with each preparation batch containing a matrix spike.*
  - Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within  $\pm 10\%$  of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
  - Post digestion spike addition: 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 and is required if the pre-digestion matrix

spike (low-level only for CLH) is outside of control limits. 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. The analyst and or section manager must note this situation on the final analytical report.

**E. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:**

**TABLE I**

<b>Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>								
Analytes by EPA 200.7,3005A/30 50A- 6010B SOW 4.1 & 5.2	AQUEOUS MDL (ug/L)	AQUEOUS ERL (ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
<b>Silver</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>	<b>0.20</b>	<b>2.0</b>	<b>2</b>	<b>2</b>
<b>Aluminum</b>	<b>50</b>	<b>200</b>	<b>200</b>	<b>200</b>	<b>10</b>	<b>40</b>	<b>40</b>	<b>40</b>
<b>Arsenic</b>	<b>3.0</b>	<b>10</b>	<b>10</b>	<b>15</b>	<b>0.6</b>	<b>2.0</b>	<b>2</b>	<b>3</b>
<b>Barium</b>	<b>5.0</b>	<b>200</b>	<b>200</b>	<b>200</b>	<b>1.0</b>	<b>40</b>	<b>40</b>	<b>40</b>
<b>Beryllium</b>	<b>1.0</b>	<b>5.0</b>	<b>5</b>	<b>5</b>	<b>0.20</b>	<b>1.0</b>	<b>1</b>	<b>1</b>
<b>Calcium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>20</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Cadmium</b>	<b>1.0</b>	<b>5.0</b>	<b>5</b>	<b>5</b>	<b>0.20</b>	<b>1.0</b>	<b>1</b>	<b>1</b>
<b>Cobalt</b>	<b>5.0</b>	<b>50</b>	<b>50</b>	<b>50</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>
<b>Chromium</b>	<b>2.0</b>	<b>10</b>	<b>10</b>	<b>10</b>	<b>0.40</b>	<b>2.0</b>	<b>2</b>	<b>2</b>
<b>Copper</b>	<b>4.0</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>0.40</b>	<b>5.0</b>	<b>5</b>	<b>5</b>
<b>Iron</b>	<b>30</b>	<b>100</b>	<b>100</b>	<b>100</b>	<b>1.0</b>	<b>20</b>	<b>20</b>	<b>20</b>
<b>Potassium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Magnesium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Manganese</b>	<b>1.0</b>	<b>15</b>	<b>15</b>	<b>15</b>	<b>0.20</b>	<b>3.0</b>	<b>3</b>	<b>3</b>
<b>Sodium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Nickel</b>	<b>3.0</b>	<b>40</b>	<b>40</b>	<b>40</b>	<b>1.0</b>	<b>8.0</b>	<b>8</b>	<b>8</b>
<b>Lead</b>	<b>1.5</b>	<b>5.0</b>	<b>3</b>	<b>10</b>	<b>0.60</b>	<b>2.0</b>	<b>0.6</b>	<b>2</b>
<b>Selenium</b>	<b>3.0</b>	<b>10</b>	<b>5</b>	<b>35</b>	<b>0.60</b>	<b>2.0</b>	<b>1</b>	<b>7</b>
<b>Antimony</b>	<b>5.0</b>	<b>60</b>	<b>60</b>	<b>60</b>	<b>1.0</b>	<b>12</b>	<b>12</b>	<b>12</b>
<b>Thallium</b>	<b>3.0</b>	<b>10</b>	<b>10</b>	<b>25</b>	<b>0.60</b>	<b>2.0</b>	<b>2</b>	<b>5</b>
<b>Vanadium</b>	<b>5.0</b>	<b>50</b>	<b>50</b>	<b>50</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>
<b>Zinc</b>	<b>5.0</b>	<b>20</b>	<b>20</b>	<b>60</b>	<b>1.0</b>	<b>4.0</b>	<b>4</b>	<b>12</b>

**TABLE 2**

<b>METAL</b>	<b>WAVELENGTH</b>
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

**XII. CORRECTIVE ACTIONS****A. INSTRUMENT RELATED**

1. ICV not within  $\pm 10\%$  or  $\pm 5\%$  for 200.7
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.

2. ICB not  $\pm$ MDL or within  $\pm$  3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**
  - a. Is the problem with the solution?
    - i. Reprepare
  - b. Is the problem with the calibration?
    - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.
3. Check standards not within  $\pm$  5%
  - a. Is the problem with the solution?
    - i. Repour, reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
4. CRI not within  $\pm$  20% (Internal QC, only required for CLP work).
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
5. ICSA metals not present are not less than the CRDL for that metal, **for ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL.**
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
6. ICSAB not within  $\pm$  20%
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
7. CCV not within  $\pm$  10%
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified. Note: CLH data must

always be reanalyzed back to the last compliant CCV and not qualified.

8. CCB not  $\pm$ MDL or within  $\pm$  3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**
  - a. Is the problem with the solution?
    - i. Reprepare
  - b. Is the problem with the calibration?
    - i. Apply SW846 guidance. (See Section IX-E for additional guidance). Note: CLH data must always be reanalyzed back to the last compliant CCB and not qualified.

## B. DIGESTION RELATED

1. Preparation blank not within  $\pm$  1/2 RL and  $\pm$  RL for common contaminants USACE or RL/CRDL for other or CLP
  - a. Is the problem with the instrument?
    - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  - b. Is the problem with the digestion?
    - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be redigested or the data must be qualified on the final report.
2. LCS not within control limits
  - a. Is the problem with the instrument?
    - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  - b. Is the problem with the digestion?
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

## C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm$ 20% (if both are >5X CRDL) or  $\pm$  the CRDL ( if either are <5X CRDL).
  - a. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm$ 25%( **$\pm$  20% for DOD projects**)
  - a. Is the analyte level in the sample greater than 4X the spiking level?
    - i. If yes, the spike recovery is not evaluated.
    - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.
3. When required, post digestion spike analysis recovery not within  $\pm$ 15%.

- a. The associated sample data must be qualified on the final report.
  - b. For USACE analysis by MSA is required.
4. Serial dilution analysis percent difference not within  $\pm 10\%$ 
    - a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
      - i. If no, the serial dilution data can not be evaluated.
      - iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

### **XIII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### **XIV. REFERENCES**

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B and Method 6010C*
2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 200.7; APX-B*
3. *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

Refer to SOP-431 for common environmental laboratory definitions.

### **Addendum for USEPA CLPILM 05.2**

1. The control limit for the ICSA is at 20% or  $\pm$ CRQL whichever is greater.
2. Preparation codes are required in the digestion log See SOW Exhibit B for a listing of these codes with definitions.
3. The CRQL check standard is run at the concentration of the respective CRQLs. For a listing of CRQL for this SOW see Exhibit C. Several of the metals concentration levels have changed.
4. The spiking level for CLP ILM 05.2 is at 50 ug/L for selenium. All other spike levels remain the same as in SOW ILM 04.1.
5. The CCV shall be analyzed at a different concentration then the ICV (at or near one-half of the calibration standard concentration).
6. The post digestion spike must be analyzed at 2x the indigenous level of the sample or two times the CRQL whichever is greater.
7. A Non-prepared MDL study must be analyzed and the results of this study used for MDL reporting when sample volumes are not digested.

### **CHANGES TO FORMS for SOWCLPILM 05.2**

1. Forms must be double-sided
2. A photocopy of the instrument's direct sequential readout shall be included.
3. Undiluted samples must be reported as well as diluted samples.
4. J flags are used in place of B flags when a sample has a concentration less the CRQL but greater then or equal to the MDL.
5. A D flag is used for samples reported from a dilution.
6. All results are reported down to the MDL not the IDL.
7. Preparation codes are used on form 13.

The form for method of standard additions (MSA) has been removed and all subsequent QC has move up one form number in other words form 8 is now serial dilution when it used to be the MSA form,etcetera.

<b>ANALYST DATA REVIEW CHECKLIST Sample Number(s):</b>				
<b>Batch Number(s):</b>				
<b>Method: 6010B ( ICP )</b>				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

## **APPENDIX I**

### **Preparation Method for Calibration Standards**

**DETERMINATION OF INORGANIC ANIONS  
IN WATER BY ION CHROMATOGRAPHY  
USING THE DIONEX DX-500 ION  
CHROMATOGRAPH WITH HYDROXIDE  
ELUENT AND DIONEX COLUMN AS18  
BY METHOD 300.0/ SW846 9056 GUIDANCE**

SOP NUMBER:

SOP-145

REVISION NUMBER:

6

APPROVED BY:

*Betty DeVille*  
SECTION MANAGER

*Randy D. Ward*

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE:

01/09/09

DATE OF LAST REVIEW:

01/09/09

**DETERMINATION OF INORGANIC ANIONS IN WATER BY ION  
CHROMATOGRAPH USING THE DIONEX dx-500 ION CHROMATOGRAPH WITH  
HYDROXIDE ELUENT AND DIONEX AS18 COLUMN**

**References:**

**USEPA METHOD 300.0/ SW846 Method 9056**

**I. SCOPE AND APPLICATION:**

1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes.

**PART A.--Common Anions**

Chloride	Nitrate	Fluoride	Sulfate
Nitrite	Bromide	Ortho Phosphate	

2. Single laboratory Method Detection Limit for the above analytes is listed in Tables 1A, 1B and 1C from method 300.0. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.
  - A. In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more than a 5 nS noise/drift per minute of monitored response over the background conductivity.
3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.
4. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.
5. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

**II. SUMMARY OF METHOD**

1. A small volume of sample, 50 uL for Part A is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a

system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

### III. DEFINITIONS

1. **ANALYSIS BATCH** -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices). The analysis batch must include an Initial Calibration Check Standard, and End Calibration Check Standard, Laboratory Reagent Blank, and a Laboratory Fortified Blank. Within an ANALYSIS BATCH, for every group of **ten** field samples at least one Laboratory Fortified Matrix (LFM) and either a Field Duplicate, a Laboratory Duplicate or a duplicate of the LFM must be analyzed after the tenth field sample analysis.
2. **CALIBRATION STANDARD (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
  - A. **INITIAL CALIBRATION STANDARDS** -- A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions.
  - B. **INITIAL CALIBRATION CHECK STANDARD** -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.
  - C. **CONTINUING CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after every tenth field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.
  - D. **END CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.
3. **FIELD DUPLICATES (FD)** -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
4. **INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC)** -- A solution of one or more method analytes, surrogates or other test substances used to

evaluate the performance of the instrument system with respect to a defined set of criteria.

5. **LABORATORY DUPLICATE** -- Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.
6. **LABORATORY FORTIFIED BLANK (LFB)** --An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB 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.
7. **LABORATORY FORTIFIED SAMPLE MATRIX (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM 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 LFM corrected for background concentrations.
8. **LABORATORY REAGENT BLANK (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
9. **LINEAR CALIBRATION RANGE (LCR)** -- The concentration range over which the instrument response is linear.
10. **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.
11. **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.
12. **MINIMUM REPORTING LEVEL (MRL)** -- The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.

13. **PERFORMANCE EVALUATION SAMPLE (PE)** -- A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.
14. **QUALITY CONTROL SAMPLE (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB 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.
15. **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.

#### IV. INTERFERENCES

1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes's retention times.
  - A. A direct chromatographic coelution may be solved by changing columns, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.
  - B. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluent.
  - C. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in**

**exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.

1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response ratios between the target analytes is observed.
- D. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.
- E. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.
- F. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.
- G. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate (retention time of ~9.0 min.) indicates the end of a chromatographic run. A run time of 12 minutes is recommended to allow for the proper elution of any potentially interferrant late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

## 2. SAFETY

- A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A

formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

## V. EQUIPMENT AND SUPPLIES

1. Ion Chromatograph (IC) – Analytical system complete with eluent generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500 or equivalent. (See letter from EPA to Dionex on discussion of alternate hydroxide eluent for anions. Also since hydroxide eluent cannot be run on the traditional column Dionex AS18, 4mm (P/N 060549) or equivalent should be used.
  - a. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.
  - b. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met.
    - i. When a 4 mm column is employed, the injection volume should be 50 uL.
    - ii. Comparable results can be attained using the Dionex, AS17, 4 mm column.
2. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 53946). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.
  - a. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.
3. Detector--Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in the Quality Control section of this SOP.
4. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2 or equivalent is used by Empirical Laboratories.
5. Analytical balance--Mettler Used to accurately weigh target analyte salt for stock standard preparation ( $\pm 0.1$  mg sensitivity).

6. Micro beakers -- Plastic, disposable - used during sample preparation.
7. Syringes--Plastic, disposable, 10 mL - used during sample preparation.
8. Pipets -- Pasteur, plastic or glass, disposable, graduated, 5 mL and 10 mL.
9. Bottles -- High density polyethylene ( HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions.
10. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Gelman IC Acrodisc, PN 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.

NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.

11. Autosampler PolyVials 5-mL size, with filtercaps, 250 each --Dionex cat log # 38141.
12. Shaker for use when extracting soil samples.
13. Centrifuge to aid in separation after extraction.
14. Centrifuge tubes--50 mL capacity

## **VI. REAGENTS AND STANDARDS**

1. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.
2. A system or apparatus which automatically generates the hydroxide eluent (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluent.
3. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors.

**NOTE:** Stability of standards: Stock standards for most anions are stable for at least 6 months when stored at 4 °C. Dilute working standards should be prepared monthly.

## **VII. SAMPLE COLLECTION, PRESERVATION AND STORAGE**

1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to

insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

2. Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A: Common Anions

<u>Analyte</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
<b>Nitrate-N</b>	<b>Cool to 4 °C</b>	<b>48 hours</b>
<b>Nitrite</b>	<b>Cool to 4 °C</b>	<b>48 hours</b>
<b>Ortho Phosphate</b>	<b>Cool to 4 °C</b>	<b>48 hours</b>
Sulfate	Cool to 4 °C	28 days

3. When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the addition of the EDA preservative at time of sample collection.

## VIII. QUALITY CONTROL

1. The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.
2. INITIAL DEMONSTRATION OF PERFORMANCE
  - A. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.
  - B. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within  $\pm 10\%$  of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

- C. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [ t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

- D. MDLs should be determined every 6 months or at least annually, when a new operator begins work or whenever there is a significant change in the background, or instrument response. MDL check samples are used in connection with confirming that the MDL determined is legitimate and to monitor the instrument sensitivity periodically. MDL checks are analyzed whenever a new MDL is generated and at a minimum quarterly to monitor for any shifts in sensitivity.

### 3. ASSESSING LABORATORY PERFORMANCE

- A. Laboratory Reagent Blank (LRB) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL (**For DOD QSM Ver. 3 no analytes detected  $\geq \frac{1}{2}$  RL or for common lab contaminants no analyte detected  $\geq$  RL**) indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.
- B. Laboratory Fortified Blank (LFB)—The LFB should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the LFM. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
- i. Control Limits for the LFB are 90 to 110%.
  - ii. The laboratory uses the LFB to assess laboratory performance against the required control limits listed in the QC section. When sufficient internal performance data becomes available

(usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery ( $x$ ) and the standard deviation ( $S$ ) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3S$$

$$\text{LOWER CONTROL LIMIT} = x - 3S$$

The optional control limits must be equal to or better than those listed in the QC section ( $\pm 10\%$ ). After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation ( $S$ ) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be on file and be available for review.

- i. Instrument Performance Check Solution (IPC) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard is 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluent (or when the KOH cartridge is changed) is prepared but if shifts of more than 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the IPC to provide evidence of an analytical column's vitality.

#### 4. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

- A. Laboratory Fortified Sample Matrix (LFM) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The LFM sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify

the LFM be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

- i. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.
- ii. The LFM should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the LFM must be fortified no greater than five times the lowest calibration level which as outlined in this method is the minimum reported level (MRL). For example, if chloride is not detected in any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest LFM fortified concentration allowed is 25.0 ug/L.
- iii. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.  
C<sub>s</sub> = fortified sample concentration  
C = sample background concentration  
S = concentration equivalent of analyte added to sample.

- iv. Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 80 to 120%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 80 –120%.
- v. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a “matrix induced bias” qualifier.

B. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

- i. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I<sub>c</sub>) and duplicate quantitated concentration (D<sub>c</sub>) using the following formula,

$$RPD = \frac{(I_c - D_c)}{[(I_c + D_c)/2]} \times 100$$

- ii. Duplicate analysis acceptance criteria

<u>Concentration range</u>	<u>RPD Limits</u>
MRL to 10xMRL	± 20%
10xMRL to highest calibration level	± 10%

- iii. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.

C. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

D. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.

E. The laboratory adopts additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participate in relevant performance evaluation sample studies.

5. **CALIBRATION AND STANDARDIZATION**

- A. Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1C for a 4-mm column.
- i. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.
  - ii. For an individual calibration curve, a minimum of eight calibration standards is required for a curve.
- B. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are calibrated from 0.5-200 mg/L; fluoride, nitrate, and nitrite from 0.05-20 mg/L.
- C. Using a 4mm column, inject 50 uL (Part A) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD)), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.
- i. Peak areas strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.
- D. Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard. This verification must be performed on each analysis day or whenever fresh eluent has been prepared. A continuing calibration check standard must be analyzed after every tenth sample and at the end of the analysis set as an end calibration check standard. The response for the initial, continuing and end calibration check must satisfy the QC criteria of 90 to 110%. If during the analysis set, the response differs by more than the calibration verification criteria, or the retention times shift more than  $\pm 5\%$  from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.

- i. Control limits for calibration verification are 90 to 110%.
- F. After satisfying the requirements, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.

6. **PROCEDURE**

- A. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.
- B. Check system calibration daily and, if required, recalibrate as necessarily.
- C. Sample Preparation
  - i. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.
- D. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 um particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.
- E. Using a 4 mm column, inject 50 uL of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.
- F. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.
- G. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to  $\pm 25\%$  the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required.

- H. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.
- I. Should more complete resolution be needed between any two coeluting peaks, the eluent can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.
  - i. Eluent dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely effect the MDLs for each analyte.

## 7. DATA ANALYSIS AND CALCULATIONS

- A. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.
- B. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).
- C. Report results for Part A anions in mg/L.
- D. Report  $\text{NO}_3^-$  as N

### Traceability

A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, date opened, and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded.

There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

## **IX. INSTRUMENT INFORMATION**

Analyst should confirm the following:

### **1. Start Up routine for instrument is as follows:**

- a. Turn Power on to autosampler, conductivity detector, eluent generator and pump in any order.**
- b. Turn on the Helium gas supply~ 80 to 100. Ensure gas lines to bottles not in use are off. Ensure air supply to the injection valve is on.**
- c. Close the vent valves on sparging bottles and allow head pressure to build for a few moments. DI H<sub>2</sub>O bottles should be 7 to 10 psi.**
- d. Open the eluent supply valve(s) for the bottles in use. Check for sputter after eluent flow has started.**
- e. Load the autosampler cartridges and put autosampler into RUN state.**
- f. To vent airlocks, Run eluent with pump on and SRS off, open waste valve-> bottom door top black knob, just for a few seconds and close it.**
- g. Turn on the SRS power. NOTE: NEVER TURN ON THE SRS POWER SUPPLY WITHOUT THE PUMP GOING FIRST!!! Use either LOCAL/DIRECT CONTROL to enter commands at keypads, or REMOTE/DIRECT CONTROL to use the direct control option from the RUN menu within PEAKNET.**
- h. After System has come to equilibrium, load sequence and run.**

### **2. Shut Down routine for instrument is as follows:**

- a. If the instrument is not going to be operated for a period of time, run deionized water through the eluent lines for ~ 30 minutes to an hour to rinse the lines.**
- b. Stop the OFF/ON pump and then select SRS-OFF.**
- c. Close gas supply valves and eluent valves on the eluent bottles. Turn off He supply. Is not necessary to vent the eluent bottles.**
- d. Power down the modules in any order.**

### 3. General Sample Loading and Run Set-up.

- a. Enter Peak-net Software from Desktop.
- b. Loading a Run: Click on Schedule. The headings within the Schedule Editor are SAMPLE, SAMPLE TYPE, LEVEL, METHOD and DATA FILE.
  - i. Name each sample under the SAMPLE heading column. (ICB, ICV, LCSW, ICCS, IPC, sample #'s, etc.)
  - ii. SAMPLE TYPE is sample unless loading a calibration curve.
  - iii. LEVEL designations are used only when assigned to a calibration curve.
  - iv. Enter method name under METHOD heading. In most cases, date of most recent calibration in Anions Method file will be used.
  - v. Enter the date under the DATA FILE heading. The program will then sequentially assign the data file names based on the date.
  - vi. All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.
  - vii. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. It is not necessary to include a vial in the corresponding position in the autosampler. Sample type is Sample, and Method is entered as <pumpoff.met>.

#### Typical run-log:

1	Blank
2	RLS@ 0.05/0.05ppm
3	ICV @ 2.5/25ppm
4	ICB
5	RLS @0.5ppm SO <sub>4</sub>
6	LCSW ISA.....
7	Sample (Cl, SO <sub>4</sub> ....)
8	Sample (Cl, SO <sub>4</sub> ....)
9	Sample (Cl, SO <sub>4</sub> ....)
10	Sample (Cl, SO <sub>4</sub> ....)
11	Sample @ 10X(Cl)
12	Sample @ 50X(Cl)
13	CCV @ 2.5/2ppm
14	CCB
15	Sample (Cl, SO <sub>4</sub> ....)

16 Sample MS  
17 Sample MSD  
18 Sample (Cl, SO<sub>4</sub>....)  
19 Sample @ 10X (Cl)  
20 Sample (Cl)  
21 Sample (Cl, SO<sub>4</sub>....)  
22 Sample (Cl, SO<sub>4</sub>....)  
23 CCV @ 2.5/25ppm  
24 CCB  
25 Sample (Cl, SO<sub>4</sub>....)  
26 Sample (Cl, SO<sub>4</sub>....)  
27 Sample (Cl, SO<sub>4</sub>....)  
28 Sample (Cl, SO<sub>4</sub>....)  
29 Sample (Cl, SO<sub>4</sub>....)  
30 Sample MS  
31 Sample MSD  
32 Sample (Cl, SO<sub>4</sub>....)  
33 CCV @ 5.0/50ppm  
34 CCB  
35 Sample (Cl, SO<sub>4</sub>....)  
36 Sample (Cl, SO<sub>4</sub>....)  
37 Sample (@ 2X ( SO<sub>4</sub>)  
38 CCV @ 5.0/50ppm  
39 CCB  
40 pumpoff

- c. Save a schedule under File/Save as, using the date as the title of the Schedule.
- d. When saved, exit out of Schedule Editor.
- e. Load autosample cartridges in the same order as the scheduled run. After putting the cartridges in the autosampler, switch the autosampler to RUN using the Hold/Run button.
- f. Assuming that the Pump is equilibrated with steady eluent baseline/uniform conductivity, enter into the RUN page.
- g. Go to File to Open Method. Open correct method <date> of most recent calibration. This will begin pumping eluent at 1.0 mL/minute and turn on the SRS pump at 300  $\mu$ amps voltage.
- h. Next, go to file to Open Schedule. Open newly created schedule for the day.
- i. When Method and Schedule are opened, go to Run and click on Start. The autosampler will inject into the first sample and the run should continue until completion.

## **X. POLLUTION PREVENTION**

- A. 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 EPA has established a preferred 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.
- B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- C. 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.

## **XI. WASTE MANAGEMENT**

- A. The Environmental Protection Agency requires that laboratory waste management practices be conducted 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 discharge permit and regulations, and by complying with all solid and hazardous 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 at the address listed in Section 14.3 from method 300.1.

## **XII. CORRECTIVE ACTIONS**

### **A. INSTRUMENT RELATED**

- 1. ICV not within  $\pm 10\%$ 
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration. Recalibrate thru analysis of appropriate standards and recheck ICV.
- 2. CCV not within  $\pm 10\%$

- a. If the problem is with the solution.
    - i. Reprepate, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and reprepate/reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalysed.
3. CCB not  $> \pm$  MDL (USACE)(For DOD QSM Ver. 3 no analyte detected  $> 2x$ MDL, frequency- beginning and ending a run and every 10 samples) or  $\pm$  RL or CRDL for others and CLP
- a. If the CCB is biased high.
    - i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.
    - ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.
  - b. If the CCB is biased low.
    - i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
    - ii. All other samples must be reanalyzed after the problem is corrected.
4. LCS not within our in-house generated control limits ( or  $\pm 10\%$  ).
- a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-extracted or the data will be qualified on the final report.

### C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within  $\pm 20\%$ 
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

### XIII. SOURCES/REFERENCES:

1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18<sup>th</sup> Edition of Standard Methods (1992).
2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.
3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December, 1981.
4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).
5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.
6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.
7. USEPA Methods 300.0; *Method for Determination of Inorganic Substances*(EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).
8. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: EPA 300.0 Anions by Ion Chromatography</b>
Instrument is a Dionex DX-500 system. Equipped with Guard Column, Analytical Column, Conductivity Suppressor, Conductivity Detector, and Eluent Generator.

<u>QA/QC Item</u>	<u>Yes</u>	<u>No</u>	<u>NA</u>	<u>Second Level Review</u>
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did RLS meet control limits?	_____	_____	_____	_____
6. Did LCS, Laboratory Fortified Blank or blank spike meet control limits?	_____	_____	_____	_____
7. Did MS/MSD meet control limits?	_____	_____	_____	_____
8. Was the Blank below the project required detection limits?	_____	_____	_____	_____
9. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
10. Were samples analyzed for Nitrate done within the 48-hr holding time?	_____	_____	_____	_____
11. Sample preparation information is correct and complete.	_____	_____	_____	_____
12. Were all samples filtered through a 0.45µm filter?	_____	_____	_____	_____
13. Analytical results are correct and complete.	_____	_____	_____	_____
14. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
15. Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
16. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____

**ANALYST DATA REVIEW CHECKLIST**  
**EPA 300.0 (Anions by IC)**

17. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete. \_\_\_\_\_

Comments on any "No" response:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

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**SULFIDE  
METHOD 376.1 and STANDARD  
METHODS SM4500S F(19<sup>th</sup> ED)  
(TITRIMETRIC, IODINE)  
WITH SAMPLE PRETREATMENT TO  
REMOVE INTERFERING  
SUBSTANCES OR TO CONCENTRATE  
THE SULFIDE**

---

**SOP NUMBER:**

**SOP-153**

---

**REVISION NUMBER:**

**3**

---

**APPROVED BY:**

*Betty DeVillo*  
SECTION MANAGER

---

*Randy Ward*  
TECHNICAL DIRECTOR

---

**EFFECTIVE DATE:**

**06/24/08**

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**DATE OF LAST REVIEW:**

**05/27/09**

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**Empirical Laboratories, LLC**

**SULFIDE**  
**METHOD 376.1 and STANDARD METHODS SM4500S F(19<sup>th</sup> ED)**  
**(TITRIMETRIC, IODINE)**  
**WITH SAMPLE PRETREATMENT TO REMOVE INTERFERING**  
**SUBSTANCES OR TO CONCENTRATE THE SULFIDE**

**I. SCOPE OF APPLICATION:**

- A. This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.
- B. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class.)
- C. This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.

**II. SUMMARY OF METHOD:**

Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate.

**III. SAMPLE HANDLING AND PRESERVATION**

- A. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.
- B. Samples are taken in glass bottles with stopper; preferably 500 to 1000 mL. Usually we use 300 mL (BOD bottle). Preserve with zinc acetate 2N and 6N NaOH. There should be no air-space in the container.
- C. The holding time for sulfides preserved in this manner is 7 days.

**IV. INTERFERENCES:**

The iodometric method suffers interferences from reducing substances that react with iodine, including thiosulfate, sulfite and various organic compounds, both solid and dissolved. Interferences due to sulfite, thiosulfate, iodide and many

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other soluble substances are eliminated by first precipitating ZnS in the samples, removing the supernatant, and replacing it with distilled water.

**V. EQUIPMENT/APPARATUS:**

- A. 10 mL burette
- B. Glass, stoppered bottle of 500 to 1000 mL (300 mL BOD bottles)
- C. Vacuum pump
- D. Magnetic stirrer with Teflon coated stirring bars
- E. Buchner funnel
- F. 500 mL Erlenmeyer flask

**VI. REAGENTS:**

- A. Zinc acetate, 2N: dissolve 220 grams Zn (C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> · 2 H<sub>2</sub>O in 870 mL D.I. water; this makes 1 liter solution.
- B. Sodium Hydroxide, 6N: dissolve 240 grams of NaOH in about 600 mL of D.I. water. Dilute to 1 liter.
- C. Hydrochloric acid, 6N: add 250 mL concentrated HCl to 250 mL D.I. water, mix well.
- D. Standard iodine solution, 0.025N: dissolve 20 to 25 grams KI in a little water and add 3.2 grams iodine. After iodine has dissolved, dilute to 1000 mL in a volumetric flask. Standardize against 0.0250N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, using thyodene as indicator. **(Purchased commercially)**
- E. Standard sodium thiosulfate titrant solution, 0.025N: dissolve 6.205 grams Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O in distilled water. Add 1.5 mL 6N NaOH or 0.4 grams solid NaOH and dilute to 1000 mL. Standardize with bi-iodate solution. (May be purchased commercially.)
- F. Standard potassium bi-iodate solution, 0.0250N: dissolve 812.4 mg KH(IO<sub>3</sub>)<sub>2</sub> in distilled water and dilute to 1000 mL. Standardization of thiosulfate--dissolve approximately 2 grams KI, free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6N H<sub>2</sub>SO<sub>4</sub> or a few drops of concentrated H<sub>2</sub>SO<sub>4</sub> and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding 1 scoop thyodene toward end of titration, when a pale straw color is reached. When the solutions are of equal strength, 20.00 mL 0.0250N Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> should

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be required. If not, adjust the  $\text{Na}_2\text{S}_2\text{O}_3$  solution to 0.0250N using  $N_1V_1 = N_2V_2$  equation.

G. Thyodene.

**VII. PROCEDURE**

A. Pretreatment:

1. If sample has not been preserved with zinc acetate and NaOH, place 3 to 5 pasteur pipettes of 2N zinc acetate solution into a 500 mL glass bottle, fill with sample, and add 10 drops 6N NaOH solution. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously about a transverse axis. Vary volume of reagents added according to sample so that the resulting precipitate is not excessively bulky and settles readily. Add enough NaOH to produce a pH above 9.
2. Let precipitate settle for 30 minutes. The treated sample is relatively stable and can be held for several hours. However, if much iron is present, oxidation may be fairly rapid. Holding time for this sample is 7 days.

B. Preparation and Titration of Sample:

1. Mark meniscus of sample volume on side of bottle so you can measure sample volume. Filter precipitate through glass fiber filter paper 11.0 to 12.5 cm in a Buchner funnel. Save the filter and all precipitate and discard filtered sample.
2. Measure exactly, amount of standard iodine solution (estimated to be an excess over the amount of sulfide present in the sample--usually 2 to 10 mL) into a 500 mL Erlenmeyer flask. Add distilled water, if necessary, to bring volume and iodine solution to 20 mL.
3. Add 2 mL 6N HCl.
4. Place filter with precipitate, making sure you wipe sides of Buchner funnel to get any precipitate clinging to the sides, into bottle with iodine solution and acid. Add 200 mL D.I. water.
5. Fill a 50 mL Burette with 0.0250 N sodium thiosulfate solution.

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6. Put a small stirring bar in sample and place on magnetic stirrer. Stir bar turning slowly.
7. Titrate slowly with sodium thiosulfate titrant adding 1 scoop thyodene reagent toward the end of the titration, when a pale straw color is reached. The sample will go from straw yellow to a dark blue when thyodene is added. It should take only 2 or 3 drops of titrant to bring sample to the endpoint of clear at this point. Record mL of titrant used. Sample will turn back blue but the first change from blue to clear is the endpoint.
8. Do a blank with each set of samples taken D.I. water through the entire procedure the same as the samples including pretreatment. D.I. water is usually ~1.0 mg/L sulfide.
9. Discard titrated sample. Rinse out bottle and measure volume of sample used by filling bottle to the calibration mark on the side of bottle with water and pouring water into a graduate cylinder.

**VIII. CALCULATIONS:**

One milliliter 0.0250N iodine solution reacts with 0.4 mg S<sub>2</sub>-:

$$\text{Mg S}_2\text{-/L} = \frac{(AXB) - (CXD)}{\text{Sample}} \times 16000 \times (\text{Ratio of final to mL initial volume})$$

where:

- A = mL standard iodine solution
- B = normality of iodine solution
- C = mL Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution
- D = normality of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution
- Final volume = 200 mL
- Initial volume = measured volume of original sample

**IX. QUALITY CONTROL:**

- A. Analyze a duplicate and second source check with each analytical batch. The second source check standard can be purchased commercially.

**X. CORRECTIVE ACTIONS**

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- A. If the LCS fails (exceeds 80-120 %). Contact Lab supervisor.

**XI. HEALTH AND SAFETY**

- A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
- B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- B. MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

**XII. POLLUTION PREVENTION**

- A. 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 EPA has established a preferred 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.
- B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- C. 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.

**XIII. WASTE MANAGEMENT**

**Empirical Laboratories, LLC**

- A. The Environmental Protection Agency requires that laboratory waste management practices be conducted 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 discharge permit and regulations, and by complying with all solid and hazardous 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 at the address listed in Section 14.3 from method 300.1.

**XIV. METHOD PERFORMANCE**

See Methods 376.1 and Standard Methods SM4500S F.

**TOTAL ALKALINITY , CARBONATE,  
BICARBONATE**

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**METHOD  
USEPA 310.1, SM2320B**

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**SOP NUMBER:** SOP-154

**REVISION NUMBER:** 5

**APPROVED BY:**

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**TECHNICAL DIRECTOR**

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## **TOTAL ALKALINITY, CARBONATE, BICARBONATE Method EPA 310.1 & Standard Methods 2320B**

### **I. SCOPE AND APPLICATION**

- A. This method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes. Soils are leached 10 grams to 100 mLs and the analysis performed on the leachate.
- B. The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 10 mL.

### **II. SUMMARY OF METHOD**

An unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The calculation for total alkalinity (in calculation section of this SOP) is then used to calculate.

When the sample is being analyzed for phenolphthalein alkalinity, carbonate, bicarbonate and total alkalinity, method 2320B is used. With this method, an unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample is then titrated to a pH exactly 0.3 pH units lower and the calculation for 2320B (in calculation section of this SOP) is used to calculate the samples for phenolphthalein alkalinity, total alkalinity, carbonate and bicarbonate results. See note at the end of section VIII (Procedure) after step N for samples with pH greater than 8.3.

### **III. DEFINITIONS**

1. **Preparation Blank (PB)**- Laboratory reagent water that is treated exactly as a sample including exposure to all glassware, equipment (pH probe) and reagents that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.
2. **Laboratory Control Sample (LCS)**- An aliquot of reagent water or other blank matrices to which known quantities of the method analyte is added in the laboratory. The LCS 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. The LCS is given a

unique identifier so that it is traceable to its source and concentration and expiration date.

3. **Analysis Batch-** An analysis batch is a group of twenty field samples, a preparation blank, a laboratory control sample and a sample and/or laboratory control sample duplicate.
4. **Sample Duplicate-** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of sample one and sample two indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.
5. **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.
6. **Performance Evaluation Sample (PE)-** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

#### IV. SAMPLE HANDLING AND PRESERVATION

- A. No preservation necessary except to keep chilled to 4°C until sample is analyzed. Do not open sample bottle until analysis.
- B. The holding time for these samples is 14 days. Example: If sampled on November 1 at 10 a.m., analysis must be performed by November 14 at 10 a.m.

#### V. INTERFERENCES

- A. Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
- B. For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in *Annual Book of ASTM Standards*, Part 31, "Water," p. 115, D-1067, Method D (1976).

- C. Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.

## VI. EQUIPMENT/APPARATUS

- A. pH meter that uses a glass electrode and can be read to 0.01 pH units. The analyst will note on the data which pH meter (either the Corning 240 or Orion 420A) is used.
- B. 50 mL disposable beakers with wide enough mouths to allow room for burette tip and pH probe.
- C. 10 mL Class A microburette.

## VII. REAGENTS

- A. Sodium carbonate solution, approximately 0.05N: Place  $2.5 \pm 0.2$  g (to nearest mg)  $\text{Na}_2\text{CO}_3$  (dried at  $250^\circ\text{C}$  for 4 hours and cooled in desiccator) into a 1-liter, Class A, volumetric flask and dilute to the mark. The  $\text{Na}_2\text{CO}_3$  solution must be disposed of after one week.
- B. Standard Acid (sulfuric or hydrochloric), 0.1N (high titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and kept on file when a purchased solution is used.** Dilute 3.0 mL concentrated  $\text{H}_2\text{SO}_4$  or 8.3 mL concentrated HCl to 1 liter with distilled water. Dilute 40 mL of 0.05N  $\text{Na}_2\text{CO}_3$  solution to 100 mL with deionized water and titrate potentiometrically with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). This standardization must be done at least every three months. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where: A = g  $\text{Na}_2\text{CO}_3$  weighed into 1 liter  
B = mL  $\text{Na}_2\text{CO}_3$  solution  
C = mL acid used to inflection point

- C. Standard Acid (sulfuric or hydrochloric), 0.02N (low titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and**

**kept on file when a purchased solution is used.** Dilute 200.0 mL of 0.1000 N Standard Acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 mL 0.05N Na<sub>2</sub>CO<sub>3</sub> solution every three months as above.

## VIII. PROCEDURE

- A. Write down time test started.
- B. Fill 10 mL microburette with Standard Acid.
- C. Pick titrant according to estimated total alkalinity. For example, a drinking water or groundwater sample would probably use the 0.020 N titrant and a wastewater sample would probably use the 0.10 N titrant. Historical data is very useful for this.

A sample size of 25 mL is usually appropriate. If you use less than 1 mL of your high titrant, then you must titrate a new sample using low titrant. Using less than 1 mL of your low titrant is valid. When the samples are soils a 12 gram portion diluted to 120 mLs is used.

- D. Sample size should be such that a sufficiently large volume of titrant is used (1 to 10 mL titrant).
- E. Standardize and calibrate the pH meter according to laboratory procedures as explained. Using the Corning 240 pH meter, first calibrate the meter by putting the probe (which has been filled with the correct filling solution) in 7 buffer, setting the pH at 7; then in 4 buffer, setting the pH at 4, and then checking 7 again to make sure it still reads 7, if an adjustment is made to the 4 buffer then check that buffer again as well. The calibration buffers must be within  $\pm 0.05$  pH units of the true value. Then check the 10 buffer. The reading should be within  $\pm 0.10$  pH units. If not, recalibration is necessary. Record this information in the appropriate log book. If automatic temperature compensation is not provided, make titration at  $25 \pm 2^\circ\text{C}$ . Check the buffer every 3 hours after calibration. The reading should be within  $\pm 0.20$  pH units.
- F. Carefully pour 25 mLs into disposable beaker by gently pouring down the side of the vessel so as to have the least aeration to sample as possible. Place a small magnetic stirring bar in vessel and start magnetic stirrer at medium to slow stirring.

Note 1: When soils are being analyzed 12 grams is weighed into a 120 mL bottle and diluted to 120 mLs. Place in the shaker for one hour. Mix the

sample well and use 25 mLs to analyze. Make sure you get a representative sample for analysis.

Note2: Where sample volume is adequate when using low titrant, a sample volume of 100 to 200 mL should be used and titration should be performed using a 10 mL microburette.

- G. Place pH probe (which has been rinsed with DI water and patted dry with a Kimwipe) in the sample such that the probe tip is not touching the sides or bottom of the flask or beaker. If the probe has a protective cover, this is not a consideration.
- H. Make sure there are no air bubbles at the bottom of filled burette. Wipe tip of burette so that no extra drops are clinging to it. Place tip of burette into mouth of vessel so that it is above the surface of the sample but is not touching the sides of the flask and drops can go nowhere but into the sample (e.g., drops from burette are not going onto pH probe or walls of flask but directly into sample).
- I. Titrate a blank and an LCS first. This will let you know that the normalities of titrant are correct. If the result is out of the acceptable range of the LCS, run a duplicate LCS. If still out of range, find another second source and if still incorrect, restandardize titrant. First double-check titrant normality.
- J. Record sample pH after reading is stable for 5 to 10 seconds.
- K. Titrate sample to pH 4.5. This must be done slowly so as not to miss the exact pH. Record titrant volume.
- L. The minimum titrant volume to be employed using high titrant is 1 mL. If high titrant is being used, go to low titrant; if low titrant doesn't work, use more sample. Be aware of sample volume that may be needed for other analyses. Do not dilute.
- M. When titrating the sample, be sure to allow time for the pH to equilibrate so that the inflection point will not be passed.
- N. Place pH probe in 7 buffer between samples. If this does not read 7, recalibrate between 4 and 7.
- O. Potentiometric titration of low alkalinity
  - a. For alkalinity of < 20 mg/L titrate 100 – 200 mL as above using a 10 mL microburet and 0.02 N acid solution.

- b. Stop titration at pH in range of 4.3-4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume. See note below.

Note : For method 2320B, if the pH of the sample is above 8.3, the sample needs to be titrated for phenolphthalein alkalinity, first check original pH. If it is not above 8.3, the phenolphthalein result will be below the minimum detection limit. If the original pH is above 8.3, titrate sample as in above procedure but down to 8.3 instead of 4.5, and record titrant volume in box A on the alkalinity bench sheet. Then proceed with the regular procedure titrating to pH 4.5, and recording this result in box B on the alkalinity bench sheet, then carefully titrate exactly 0.3 pH units lower to pH 4.2 and record titrant volume at this level, in box C on the alkalinity bench sheet.

- P. Potentiometric titration of high alkalinity: Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp endpoint.
  1. For >1000 mg CaCO<sub>3</sub>/L use 0.1 N titrant
    - i. For alkalinity of > 1000 mg CaCO<sub>3</sub>/L, titrate 25 – 50 mL as above using a 50 mL burette and 0.10 N acid solution.
    - ii. Stop titration at pH in range of 4.5, record volume and exact pH. See note above.

## IX. CALCULATIONS

The detection limit is 1.0 mg/L CaCO<sub>3</sub>.

Potentiometric titration to pH 4.5 (high alkalinity)

$$\text{Total Alkalinity mg/ L CaCO}_3 = \frac{A \times N \times 50,000}{\text{mL of Sample}}$$

Where: A = mL Standard Acid to pH 4.5  
N = Normality Standard Acid

Potentiometric titration of low alkalinity = T below

Method 2320B calculations

Use the following notation in below calculations:

P = Phenolphthalein alkalinity

T = Total alkalinity

$$P = \frac{A \times N \times 50,000}{\text{mL of Sample}}$$

$$T = \frac{(2B-C) \times N \times 50,000}{\text{mL of Sample}}$$

mL of Sample		mL of Sample	
If P = 0	Carbonate = 0	Bicarbonate = T	
If P < 1/2T	Carbonate = 2P	Bicarbonate = T-2P	
If P = 1/2T	Carbonate = 2P	Bicarbonate = 0	
If P > 1/2T	Carbonate = 2(T-P)	Bicarbonate = 0	
If P = T	Carbonate = 0	Bicarbonate = 0	

Where: A = mL titrant to pH 8.3  
B = mL titrant to pH 4.5  
C = mL titrant to pH 4.2  
N = normality Standard Acid

## X. QUALITY CONTROL

- A. Run a laboratory control sample (LCS) for each batch of samples (maximum of 20 samples per day). If the LCS does not fall in the range of 80 to 120%, corrective action must be taken to find the problem and correct it.
- B. Run a preparation blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than the reporting limit.
- C. Analyze a sample duplicate every 10 to 20 samples (depending on specific client requirements). Relative percent difference (RPD) on duplicates should be less than 20%.
- D. The Excel file for calculations is located in "V:\Wetchem\TESTS\CarbonDioxide Alkalinity\".
- E. Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

## **XI. CORRECTIVE ACTION**

- A. If the preparation blank is higher than the reporting limit, all samples less than ten times the concentration of the blank must be reanalyzed.
- B. If the laboratory control sample (LCS) is out of the range of 80 to 120%, and the % recovery is high (higher than 120%), only sample concentrations less than the reporting limit are acceptable data. Otherwise, all data with concentrations above the method detection limit must be reanalyzed with an LCS in the range of 80 to 120%. If the LCS is low (less than 80%), all samples must be reanalyzed.
- C. If the relative percent difference (RPD) between the sample and the sample duplicate are out of the range of 20%, the sample should be repeated one more time to make sure that the problem is not caused by analyst's error. If the RPD is still higher than 20% the sample is flagged on the final report with a "\*".

## **XII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **XIII. SAFETY**

- A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

## **XIV. METHOD PERFORMANCE**

- A. Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/L, CaCO <sub>3</sub>	Precision as Standard Deviation mg/L, CaCO <sub>3</sub>	Accuracy as	
		Bias, %	Bias, mg/L, CaCO <sub>3</sub>
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

## XV. REFERENCES

- A. Methods for the Chemical Analysis of Water and Wastes, EPA Series Method 310.1.
- B. Standard Methods for the Examination of Water and Wastewater, 14<sup>th</sup> Edition, p.278, method 403, (1975).
- C. Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, p. 2-26, method 2320B, (1992).

**DISTILLATION OF AQUEOUS/SOLID  
SAMPLES FOR TOTAL AND NON-AMENABLE  
CYANIDE ANALYSIS**

**METHODS 335.1/335.4/Standard Methods SM4500-  
CN C,G, 18<sup>th</sup>, 19<sup>th</sup> ED./ (SW846) 9012A/USEPA CLP  
ILMO 4.1  
(NJDEP does not accept CLPILM 04.1 after June,  
2003)  
Addendum for USEPA CLPILM 05.2 AQUEOUS  
&SOIL/SEDIMENT**

**SOP NUMBER:**

**SOP-164**

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**APPROVED BY:**

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**03/09/09**

**DATE OF LAST REVIEW:**

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## DISTILLATION OF CYANIDE, TOTAL AND AMENABLE

### REFERENCE:

SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18<sup>th</sup>, 19<sup>th</sup> ED. / CLP ILMO 4.1

See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

### I. SCOPE AND APPLICATION

- A. This method is applicable to the distillation of cyanide from drinking, surface, and saline waters, domestic and industrial wastes, and soil/sediments.
- B. The limit of detection for waters is 0.0050 mg/L and the limit of quantitation is 0.010 mg/L. The limit of detection for soils is 0.13 mg/kg and the limit of quantitation is 0.25 mg/kg.

### II. SUMMARY OF METHOD

- A. The cyanide as hydrocyanic acid is released from cyanide complexes by means of a reflux distillation operation, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.
- B. Cyanide is defined as cyanide ion (CN<sup>-</sup>) and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

### III. SAMPLE HANDLING AND PRESERVATION

- A. The sample should be collected in plastic or glass bottles of 100 mLs. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.
- B. Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.06 g of ascorbic acid for each liter of sample volume.

- C. Samples (not including soils/sediments) must be preserved with 2 mL of ten normal sodium hydroxide per liter of sample ( $\text{pH} \geq 12$ ) at time of collection.
- D. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator to maintain temperature at 4°C.
- E. The holding time for cyanide is 14 days counting the day of sampling as the first day. This applies for soils as well as waters. Samples must be distilled and analyzed within 14 days of sampling.

#### IV. INTERFERENCES

- A. Interferences are eliminated or reduced by using the distillation procedure.
- B. Sulfides adversely affect the colorimetric procedures. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation, must be treated by adding lead acetate, or if the sulfide concentration is too high, add powdered lead carbonate  $[\text{Pb}(\text{CO}_3)_2]$  to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Filter sample before raising pH for stabilization.
- C. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. Nitrate and nitrite interference is eliminated by adding 0.5 ml of sulfamic acid solution (VII.A.7) before distillation.
- D. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser.

#### V. SAFETY

- A. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions.**
- B. If an alkaline solution containing simple cyanide is acidified, the simple cyanide will be released.
- C. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential

health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

- D. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

## VI. EQUIPMENT/APPARATUS

- A. Reflux distillation apparatus such as shown in Figure 1 of the method. Midi distillation apparatus which uses Hammett Scientific Glassware Note: This glassware is made to fit our specific block digester for cyanide. Glassware orders must be made in advance since they are not made until ordered.

## VII. REAGENTS AND STANDARDS PREPARATION

### A. Reagents

1. **Sodium Hydroxide, 0.25N:** Dissolve 10 g grams of NaOH in DI water and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.
2. **Sulfuric Acid 1:1:** Slowly add 500 mL of concentrated H<sub>2</sub>SO<sub>4</sub> to 500 mL DI water. Use caution because solution will become extremely hot!! Allow to cool while continuing to stir. Store in a labeled container at room temperature.
4. **Magnesium Chloride Solution:** Weigh 510 g of MgCl<sub>2</sub>•6H<sub>2</sub>O into a 1 liter flask; dissolve and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.
5. **Chlorine Bleach Made with Sodium Hypochlorite**, such as Chlorox.
6. **Rhodanine Indicator:**  
Dissolve 20 mg of p-dimethyl-amino-bensalrhodanine in 100 mL acetone.

7. **Sulfamic Acid:** Dissolve 40 g of sulfamic acid in DI water, dilute to 100 mL (saturated solution). **NOTE:** When making this reagent for use with the maxi-distillation system the 40 g of sulfamic are diluted to one liter with DI water
8. **Phenolphthalein solution:** Commercially prepared.
9. **Lead Acetate Test Paper:** Commercially prepared.
10. **Acetate Buffer Solution, pH 4.0:** Dissolve 146g anhydrous  $\text{NaC}_2\text{H}_3\text{O}_2$ , or 243 g  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ , in 400 mL distilled water, add 480 g conc. acetic acid, and dilute to 1 L with chlorine-demand-free water.
11. **Lead Acetate**
12. **Powdered Lead Carbonate  $[\text{Pb}(\text{CO}_3)_2]$**

## B. Standards

### 1. Traceability

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

### 2. Preparation

- a. Stock Cyanide Solution at 1000 mg/L: Commercially prepared.
- b. Working Standard Cyanide Solution: Prepare fresh daily by diluting 1 mL of stock cyanide solution to 100 mL in a volumetric flask with using 0.25N NaOH as diluent. 1 mL - 10 µg CN. Record the preparation in the log book.
- c. Performance Evaluation (PE) samples which we have analyzed and received true values for are used for the Laboratory Control Samples. These PE samples are given a unique identifier and all information such as expiration date (typically one year from the date made), source, concentration and preservation are recorded in the Inorganic Standards book A (ISA). The ISA # assigned is used to label this PE sample and is used on all data to trace it back to its source and true concentration.

## VIII. CALIBRATION

- A. Not applicable. See SOP-175 “**POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT**” for analysis.

## IX. PROCEDURE

### A. Pretreatment for cyanides amenable to chlorination

1. Two sample aliquots are required to determine cyanides amenable to chlorination. Place one 25 mL aliquot or a volume diluted to 25 mL, in a 100 mL beaker, under the hood, add a stirring bar, and place on a magnetic stirrer, and start stirring bar turning.
2. Check pH and adjust to between 11 and 12 units, with 1.25N NaOH. Use NaOH pellets if solution is highly buffered.
3. Add sodium hypochlorite chlorine bleach (Chlorox) solution dropwise until a drop of well-mixed sample turns KI starch paper a distinct blue color. Add about 10 drops excess bleach. (Caution: the initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride.) Maintain this excess for one hour, continuing agitation. Check periodically during the hour (15 minute intervals) to make sure an excess is maintained. If necessary, add additional hypochlorite bleach solution. Also check the pH and adjust back up to 11 to 12 units if necessary.

4. After one hour, add 0.025 g portions of ascorbic acid until KI starch paper shows no residual chlorine. Add an additional 0.025 g of ascorbic acid to ensure the presence of excess reducing agent.
5. Turn off stirring plate and remove stirring bar. Proceed with distillation as in the procedure.
6. Test for total cyanide in both the chlorinated and unchlorinated aliquots. (The difference of total cyanide in the chlorinated and unchlorinated aliquots is the cyanide amenable to chlorination.)

### **B. Spikes and high and low check standards**

1. **Spikes:** Matrix spikes are prepared by diluting 2.5 mL of the High DCV solution in #3 below, to 25 mL with sample (adding 22.5 mL of sample and 2.5 mL of DCV high to the distillation tube). The concentration of this spike is 0.10 mg/L.
2. **Low Distilled Check standard (low DCV).** Dilute 2.0 mL of the 10 mg/L working standard to 100 mLs in a volumetric flask. Then dilute 6.0 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.048 mg/L standard.
3. **High Distilled Check standard (high DCV).** Dilute 10 mLs of the 10 mg/L working standard to 100 mLs in a volumetric flask with DI water. Then dilute 5 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.20 mg/L standard.

### **C. Distillation**

1. For Waters: Place 25 mL of sample, or an aliquot diluted to 25 mL, in a sample tube. 0.25 N NaOH must be used for all sample dilutions following the distillation.
2. For Soils or Sludges:
  - a.) It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
- Two quarters should then be mixed to form halves.
- The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed.

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

- b.) Accurately weigh a representative 1-g minimum (record actual weight) portion of wet-weight sample (unless there are project-specific requirements where a lower detection limit is required) and transfer it to a sample tube. Add 25 mL of 0.25 N NaOH. Shake or stir the sample so that it is dispersed.
3. Add one drop of Phenolphthalein solution to verify preservation. A positive test is indicated by a bright pink color. If not add a small amount of NaOH.
  4. To test for sulfide, place a drop of sample on lead acetate paper, previously moistened with acetic acid buffer solution (pH 4). A positive test is indicated by a black color on the paper.
    - a.) If positive the sample will need to be filtered through 934AH filter paper. (4.5cm size) The filtrate will be treated and the filter added back to the treated filtrate before distillation.
    - b.) Treat filtrate with powdered lead carbonate [ $\text{Pb}(\text{CO}_3)_2$ ]. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper.
    - c.) Filter the resulting precipitate (should form a lead sulfide which will be black in appearance).
    - d.) Once the filtrate is treated and filtered, the filter with the original solids from the pretreated sample will be added back to the treated filtrate and then distilled.

5. Add 25 mL of sodium hydroxide solution 0.25N to the absorber and assemble the apparatus as shown in Fig. 1.
6. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum such that air bubbles from the thistle tube in the flask at a rate of 3 to 5 per second.

**Note:** The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally to prevent the solution in the sample tube from boiling and spewing out through the thistle tube.

7. Turn on condenser water.
8. Add 0.5 mLs of sulfamic acid through the thistle tube.
9. Slowly add 2 mL 1:1 H<sub>2</sub>SO<sub>4</sub> through the thistle tube.
10. Add 2 mL 2.5 M MgCl<sub>2</sub> through the thistle and rinse it down with deionized water.
11. Reflux at 100°C for 90 minutes, turn off heat, allow to cool with pump on for 15 minutes.
12. Disconnect absorber and transfer the solution from the absorber into a storage container.
14. Record distillation information in the distillation log. Include date, analyst, sample number, client, matrix, position, sample mass / volume, distillation time, cool down time, when transferred to analyst, when analyzed.

## X. CALCULATIONS

- A. Sample volumes / masses must be recorded accurately in the distillation log to allow the analyst to correctly calculate the final concentration of cyanide correctly.

## XI. DISTILLATION GLASSWARE CLEANING & LABELING

- A. The distillation glassware is cleaned thoroughly with hot soapy water between distillations. 1:1 HCl is aerated through the frits on the block to ensure that the frits are clean. Deionized water is forced up through the glassware to rinse it. The cold fingers are dipped in mild acid, rinsed and wiped off.
- B. All glassware must be labeled and each position's label must be recorded in the distillation log with the sample that was distilled in that particular vessel.
- C. When sample concentrations are at or exceed 1000 mg/L a deionized water blank must be distilled in the exact glassware used for that sample before processing any other client's sample to ensure that the glassware is cleaned and has no residual cyanide to carryover.

## **XII. QUALITY CONTROL**

- A. With each distillation batch of samples (maximum of 20 samples) a preparation blank (PB) must be distilled.
- B. Each distillation batch of aqueous samples **requires a laboratory control sample (LCS) each day or per 20 samples whichever is more frequent.** Under CLP the LCS serves as the distilled ICV.
- C. With each distillation batch of soil samples a soil LCS **each day or per 20 samples whichever is more frequent.**
- D. A high and low check standard ( DCV/ICV) must be distilled each day or per 20 samples whichever is more frequent. A non-distilled check standard must be prepared daily. This will be used to verify the preparation of the DCV if the DCV results are outside the specification limits. Under CLP the aqueous LCS serves as the ICV.
- E. A sample spike (MS) and sample spike duplicate (MSD) are performed with each set of twenty samples. CLP requires 1 duplicate and 1 spike per batch. All QC samples should be taken through the whole procedure including distillation. Spikes are added to the boiling flask in the same manner as standards adding 25 mL of samples or a dilution as used for the unspiked sample. Spike concentration is listed in the method. The addition of the spiking solution must be witnessed and such must be noted in the digestion log.

## **XIII. CORRECTIVE ACTION**

- A. If any problems, such as low or high recovery for check standards or LCSs occur, contact the Group Leader. Problems most frequently encountered (for the distillation procedure) are listed below.
- B. Recoveries are affected in various ways.
1. If the spiking solution is not prepared properly (e.g., making the proper dilutions), the check standard will be out of control. Caution should be used in calculating the dilution and performing the pipetting.
  2. Also, the air flow from the vacuum pumps can cause low recovery. Pay attention to the air flow (it should be in the area of 1 to 2 bubbles per second). If it is too great the HCN does not have an adequate amount of time to be absorbed by the NaOH solution.
  3. If the heating block is not heating properly, this will also lead to low recovery. During the distillation, periodically check the sample tubes for the presence of heat (tubes should be very hot to the touch).
  4. The addition of all reagents, including the spiking solution, is essential. Without the  $MgCl_2$ , 1:1  $H_2SO_4$ , or spiking solution, the percent recovery will suffer greatly for complex cyanide. The recovery of simple cyanides will usually be unaffected.
  5. If the volume of 0.25 N NaOH and reagent water is not accurate in the NaOH absorber tubes, low recoveries will usually occur.
- B. Spike recoveries are affected in similar ways. Refer to the above suggestions. Also, if possible, do not spike a sample that is known to be high in cyanide. This may cause recovery and calculation problems.

#### **XIV. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT**

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.
2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.
3. The QC criteria for both the Distillation Check QC and the distilled ICV is  $\pm 15\%$ .
4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is  $\pm 30\%$ .
5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is  $\geq 5$ , round up; otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, ( 3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

### **XV. DEFINITIONS**

See SOP-431 for a list of definitions

**POST-DISTILLATION ANALYSIS FOR**  
**CYANIDE BY THE LACHAT**  
**METHODS 335.4;(SW846) 9012A**  
**USEPA-CLP 4.1**  
**(NJDEP does not accept CLPILM 04.1 after**  
**June, 2003)**  
**Addendum for USEPA CLPILM 05.2**  
**AQUEOUS & SOIL/SEDIMENT**

**SOP NUMBER:** SOP-175

**REVISION NUMBER:** 9

**APPROVED BY:**  
Betty DeVill  
**SECTION MANAGER**

Randy D. Ward  
**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** 07/18/06

**DATE OF LAST REVISION:** 07/0708

**POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT  
0.005-0.500 mg CN-/L**

**References:**

**USEPA METHOD 335.4/(SW846) 9012A/USEPA CLP ILMO 4.1  
See Addendum for USEPA CLPILM 05.2 (Soil/Sediment)**

**I. PRINCIPLE**

Cyanide from alkaline distillates is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH lower than 8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The color is read at 570 nm.

**II. INTERFERENCES**

1. For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides (sulfides are removed with a lead acetate scrubber using the maxi distillation system) fatty acids, aldehydes and thiocyanates may distill over. See Empirical Laboratories Method SOP-164 for distillation procedure.

**III. SPECIAL APPARATUS**

1. Lachat Quick Chem AE Automated Ion Analyzer
2. Cyanide manifold (Lachat)

**IV. SAMPLE HANDLING AND PRESERVATION:**

The color reaction is pH sensitive. Therefore, distillates and standards should be carefully matched with respect to NaOH concentration. The distillates are 0.25 N NaOH.

**V. SAMPLE PREPARATION**

The samples for analysis come directly from the midi-scale distillation procedure.

**VI. PREPARATION OF REAGENTS**

Use deionized water (<10 megaohm) for all solutions.

**Degassing with helium**

To prevent bubble formation in the flow cell which can cause inaccurate readings due to airspikes (characterized by poor peak integrity factors (PIF) and/or inaccurate results) degass all solutions except the standards and the Pyridine-Barbituric Acid reagent with helium. Use Helium at 140 kPa (20 lb/in<sup>2</sup>) through a helium degassing tube (Lachat part 50100). Bubble Helium vigorously through the solution for one minute.

### **Reagent 1. Carrier, 0.25 M Sodium Hydroxide**

In a 1L vol. flask add 200 mL of 1.25N NaOH (which is used in the SOP-164 distillation method) to approximately 600 mL of D.I. water. Dilute to 1 L, invert to mix. This will ensure that the concentration of the NaOH used for the distillates will be the same concentration used for the carrier solution.

### **Reagent 2. Phosphate Buffer, 0.71 M**

In a 1 L volumetric flask, dissolve 97 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous,  $\text{KH}_2\text{PO}_4$ ) in approximately 800 mL water. Dilute to the mark and invert to mix. Prepare fresh monthly.

### **Reagent 3. Chloramine-T Hydrate**

To a 500 mL volumetric flask add about 250 mL water, then add 2.0 g chloramine-T [ $\text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{N}(\text{Cl})\text{Na} \times \text{H}_2\text{O}$ ]. Dilute to the mark and invert to mix. Prepare fresh daily. The shelf life of the chloramine-T can be a critical factor in the Lachat run. New Chloramine-T should be ordered every 6 months. (Analyst discretion should be used).

### **Reagent 4. Pyridine-Barbituric Acid Reagent**

In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100.0 mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine ( $\text{C}_5\text{H}_5\text{N}$ ) with stirring and mix until the barbituric acid dissolves. Add 15 mL concentrated hydrochloric acid (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert to mix. This reagent is stable for approximately six months if stored in a cool, dark place.

## **VII. PREPARATION OF STANDARDS**

### **Traceability**

A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

**Standard 1. 1000 mg CN/L** [~1,000 mg CN<sup>-</sup>/L (Stock)]

The Stock Standard is purchased from a vendor. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions. Use gloves and protective equipment when handling standards and samples.** This solution is given a unique identifier.

NOTE: The cyanide ion present in the original sample is concentrated by the macro-distillation procedure. The standards preparation recipes below are for standards which are not distilled. These standards are prepared in 0.25 M NaOH to match the matrix of the distilled samples. Since the samples are concentrated by a factor of two; a x 0.50 correction factor must be included in the final manual dilution factor entered into the Lachat. For example, a x 100 dilution would have a final manual dilution factor of 50 and an undiluted sample would have a final manual dilution factor of 0.50.

Record preparation in the appropriate log book.

**Standard 2. Working Standard (10.0 mg CN/L)**

Pipette 2.00 mL Standard 1 into a 200 mL volumetric flask. Dilute to the mark with Reagent 1, 0.25 M Sodium Hydroxide. Invert to mix.

Set of six calibration Standards:

10 mL of 10 mg/L diluted to 100 mL = 1.0 mg/L std  
5.0 mL of 10 mg/L diluted to 100 mL = 0.500 mg/L std  
2.0 mL of 10 mg/L std diluted to 100 mL = 0.200 mg/L std  
1.0 mL of 10 mg/L std diluted to 100 mL = 0.100 mg/L std  
4 mL of 0.500 mg/L std diluted to 100 mL = 0.020 mg/L std  
2 mL of 0.500 mg/L std diluted to 100 mL = 0.010 mg/L std  
1 mL of 0.500 mg/L std diluted to 100 mL = 0.0050 mg/L std  
Blank consists of 0.25 M NaOH  
10 mL of 10.0 mg/L std diluted to 200 mL = 0.500 mg/L chk std

The diluent for all standards is 0.25 M NaOH.

NOTE: The cyanide standards are labeled by their actual concentration as determined by standardization of the cyanide stock. For example, if the cyanide stock was determined to have a concentration of 994 mg/L (instead of 1,000 mg/L) then the "1.00 mg/L" std would actually be 0.9940 mg/L, the "0.200 mg/L" std would be 0.1988 mg/L, and so forth. Assigning values to the standards is done under the "Method Definition" option on the Lachat computer.

Record preparation in the appropriate log book.

**VIII. INSTRUMENT INFORMATION** (see page 12 for manifold diagram)

Analyst should confirm the following:

**A. Timing:**

Sample throughput: 80 samples/h; 40 s/sample  
Pump speed: 35 RPM

Cycle Period: 45 s  
Inject to start of peak period: 28 s  
Inject to end of peak period: 61 s

**B. QuikChem AE Settings:**

1) Parameter, Data Window:

Top Scale Response: 0.50 abs  
Bottom Scale Response: 0.00 abs

2) Segment/Boundaries

A - 0.50 mg CN/L  
F - 0 mg CN/L

3) Results/Approval, Reports

In the default Report definition file (RDF), change:

-Set Default Chord 0 to

-Set Default Chord 3

(Peak should be centered in chord 3)

This change must be made to both the sample and the calibration RDF's.

**C. System Notes:**

1. Allow enough time for heating unit to warm-up to 60°C with D.I. water pumping through all reagent lines at full speed. (approximately 30 min.)

**IX. CALCULATIONS**

**A. Aqueous Samples:**

The Lachat reports the concentration of the final distillate in mg/L. To correct for the X2 concentration by the distillation process, a x 0.50 correction factor must be included in the final manual dilution factor when defining a tray in the Lachat computer. For example, a x 100 dilution would have a final manual dilution factor of 50 and an undiluted sample would have a final manual dilution factor of 0.50. After the application of the X0.5 correction factor, the detection limit is 0.005 mg/L.

**B. Solid Samples:**

The Lachat reports the concentration of the final distillate in mg/L. Results for solid samples should be in mg/kg and can be obtained by entering the appropriate dilution factor when defining the tray. Conversion from mg/L to mg/kg can be accounted for by the following formula:

$$\left[ \frac{\text{mg CN}^-}{\text{Kg sample}} \right] = \left( \frac{\text{N mg}}{\text{1 L soln}} \right) \left( \frac{0.50 \text{ L soln}}{\text{g sample}} \right) \left( \frac{1,000 \text{ g sample}}{\text{1 Kg sample}} \right) (\text{Wet Weight})$$

From this formula a dilution factor can be obtained. For example if 2 grams of soil were used then this would be a x 250 dilution. When the x 0.50 correction factor is included a final manual dilution factor of x 125 is obtained.

N would be the value obtained from the Lachat without a correction factor. The minimum value for N is 0.01 mg/L soln.

C. Data reporting

1. Reduce data to the result which will be reported.
2. Complete the data review checklist ( attached ). This must be completed and attached to each set of USACE data.

X. **QUALITY CONTROL (Reference SW-846, Update III and USEPA CLP ILMO 4.1 for further clarifications)**

A. **QA/QC, distilled:**

1. PB (preparation blank)

500 mL of DI water are distilled. When brought up to 250 mL volume following the distillation process a 0.25 M NaOH solution is obtained. A blank is distilled every batch of samples. If the absolute value of the PBW is not below the CRDL or the RL **or ½ the CRDL for Navy Projects** the analyst's supervisor should be consulted before proceeding.

2. DCV (distilled calibration verification)/ICV (distilled/CLP)

a. The DCV is a check standard generally of a concentration of about 0.200 mg/L. A DCV is put on with every set of distilled samples. There is a maximum of 10 samples in a set. If the DCV is not within  $\pm 10\%$  of the undistilled value the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log. **USACE projects require both a high and low level DCV and they should compare within  $\pm 10\%$  of the undistilled standards. A corrective action report (CAR) will be required if the limit is exceeded.**

b. CLP requires the ICV to be distilled and from a second source. Must be analyzed immediately after calibration. For water samples the ICV will serve as the LCS (water only). Soils require bath. The control limit is  $\pm 15\%$ .

3. LCSW (laboratory control sample-water)

The laboratory control sample is a second source check on the calibration and must be run once every 20 aqueous samples. The control limits are in-house-generated. The CLP control limits is 80 to 120%. If the LCSW is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log.

4. LCSS (laboratory control sample-soil)

The LCSS is a second source check with a soil matrix and must be run once every 20 solid phase samples. The control limits are in-house-generated. Under CLP the agency may set the limits. If not, use in-house control limits. If the LCSS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log.

5. MS/MSD (matrix spike/matrix spike duplicate)

A spike and spike duplicate (CLP requires 1 duplicate and 1 matrix spike) are done with each batch or 20 samples. The spike concentration will usually be the same as the DCV and spikes are added to the boiling flask in the same manner as check standards. Included in the boiling flask will be the sample aliquot. The total volume for the matrix spike is 500 mL. If the percent recovery for the MS or MSD is not within  $\pm 25$  percent of the spike concentration, the analyst's supervisor should be consulted before proceeding.

6. Documentation of Capability (DOC) - Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

**B. QA/QC, undistilled:**

1. ICV (initial calibration verification)

The ICV is an undistilled check standard made from the working standard solution. The concentration is usually around the midpoint of the calibration curve (about 0.500 mg/L). The ICV is analyzed at the beginning of each Lachat run. If the ICV is not within  $\pm 10$  percent of its true value, the analyst's supervisor should be consulted before proceeding. The calibration verification standard must be prepared fresh daily.

2. ICB (initial calibration blank)

The ICB is 0.25 M NaOH solution and follows the ICV at the beginning of each Lachat run. If the absolute value of the ICB is not IRL or  $\pm$  CRDL or  $\frac{1}{2}$  the **CRDL for Navy Projects**, the analyst's supervisor should be consulted before proceeding.

3. CCV (continuing calibration verification) **From primary std. that curve is made from.**

The CCV will be analyzed after every 10 samples and at the end of the tray. The sampling of the CCV's will be done automatically by the Lachat if it is included in the tray definition. The CCV scheduling can be done under the Data Quality Management option in the Tray Definition screen. If the CCV is not within  $\pm 10$  percent of its true value a CHK STD failure signal will be given by the Lachat and the analyst's supervisor should be consulted before proceeding.

4. CCB (continuing calibration blank)

This sample verifies the instrumental baseline. If the absolute value of the CCB is not below the minimum detection limit the analyst's supervisor should be consulted before proceeding.

**C. Calibration Approval:**

For a cyanide calibration to be approved, segment (A-F) of the curve must have a correlation of at least 0.995 in chord 3.

**D. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:  
(MDLS are performed annually or whenever a change in the method is made.)**

**TABLE I**

<b>Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>								
<b>Analytes by EPA 335.1, 335.3, 9012A , SOW 4.1 &amp; 5.2</b>	<b>AQUEOUS MDL(ug/L)</b>	<b>AQUEOUS ERL(ug/L)</b>	<b>AQUEOUS CRQL ILMO 4.1 (ug/L)</b>	<b>AQUEOUS CRQL ILMO 5.2 (ug/L)</b>	<b>SOLID/SOIL MDL (mg/Kg)</b>	<b>SOLID/SOIL ERL (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)</b>
<b>Cyanide</b>	0.0050	0.005	10	10	0.007	0.13	1	1

**TABLE II**

<b>ANALYTE WAVELENGTH</b>	
<b>Cyanide</b>	<b>570.0</b>

## **XI. CORRECTIVE ACTIONS**

### **A. INSTRUMENT RELATED**

1. ICV not within  $\pm 10\%$ 
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and recheck ICV.
2. CCV not within  $\pm 10\%$ 
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalysed.
3. Ending CCB not  $\pm$  RL or CRDL **or 1/2 the CRDL for Navy Projects**
  - a. If the CCB is biased high.
    - i. Any samples BDL or greater than 5X the CCB bias need not be reanalyzed.
    - ii. Any samples above the detection limit but less than 5X the CCB level must be reanalyzed after the problem is corrected.
  - b. If the CCB is biased low.
    - i. Any samples greater than 5X the absolute CCB bias need not be reanalyzed.
    - ii. All other samples must be reanalyzed after the problem is corrected.

### **B. DIGESTION RELATED**

1. The preparation blank is not less than the  $\pm$  RL or CRDL **or 1/2 the CRDL for Navy Projects.**
  - a. If the problem with the instrument.

- i. Analyze a CCB to determine this.
    - ii. If the problem was with the instrument correct the situation and reanalyze the preparation blank.
  - b. If the problem is with the distillation.
    - i. All associated samples which are BMDL or have a level of cyanide greater than 5X the level found in the preparation blank can be reported. If the level of cyanide in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be redistilled/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.
2. LCS not within our in-house generated control limits ( or  $\pm 20\%$  ).
  - a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control.
  - b. If the problem is with the digestion.
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.
3. Distilled check standard not within control limits of  $\pm 15\%$ .
  - a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control.
  - b. If the problem is with the digestion.
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

### **C. SAMPLE MATRIX RELATED**

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$ 
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

- ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

## **XII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **XIII.SOURCES/REFERENCES:**

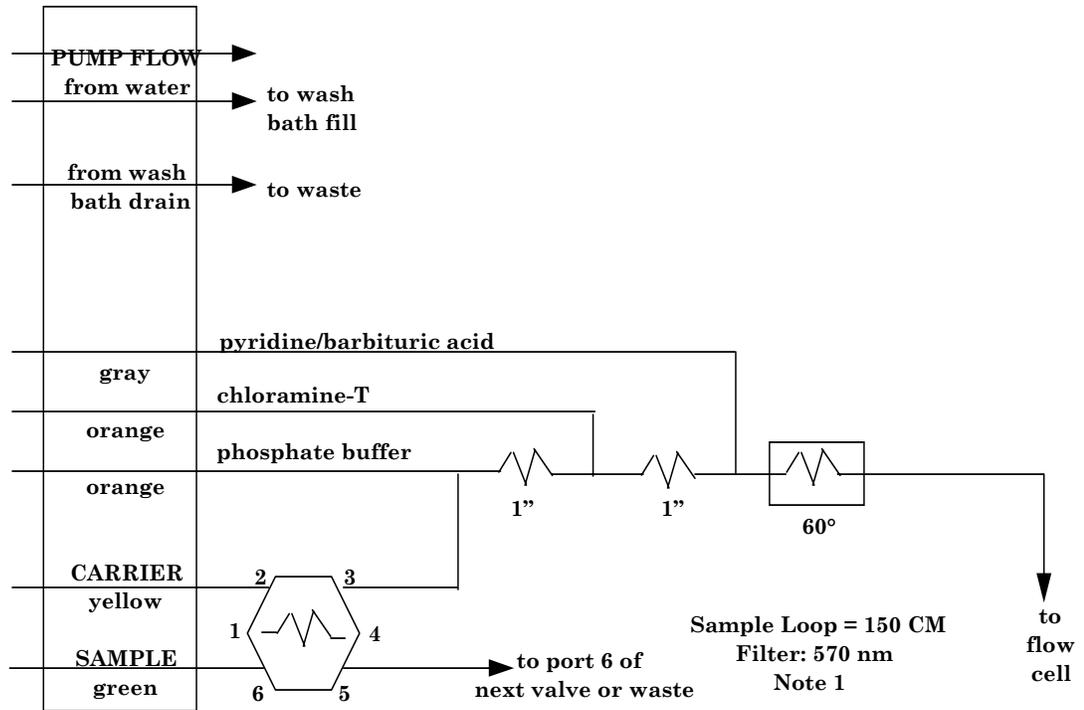
1. Lachat manual; Quick Chem method 10-204-00-1-A.
2. U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 335.4.
3. Standard Methods for the Examination of Water and Wastewater, 14th Edition, APHA-AWWA-WCPC, Park 413D, pp. 370-372.
4. SW-846, 9012A, Revision September 1986.
5. U.S. Environmental Protection Agency, C.L.P. S.O.W. ILMO 4.1.
6. QuickChem AE, Automated Ion Analyzer Training Manual, Lachat Instruments.
7. QuickChem AE, Automated Ion Analyzer Software Reference Manual, Lachat Instruments.

## **Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT**

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.
2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.
3. The QC criteria for both the Distillation Check QC and the distilled ICV is  $\pm 15\%$ .
4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is  $\pm 30\%$ .
5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is  $\geq 5$ , round up; otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, ( 3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

**MANIFOLD DIAGRAM**



CARRIER is 0.25 M Sodium Hydroxide

Note1: A 3.0 m x 0.022" id back pressure loop is placed at the exit of the flow cell.

1" is 70.0 cm of tubing on a 1 in coil support

Manifold Diagram Revision Date: 13 May 2006

APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650 cm of heated tubing.

All manifold tubing is 0.8 mm (0.032 in) i.d., This is 5.2 µL/cm.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: 9012A ( Cyanide )</b>

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was distillation temperature monitored/documentated and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

**ANALYST DATA REVIEW CHECKLIST  
9012A ( Cyanide )**

Comments on any "No" response:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

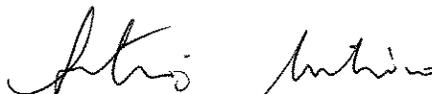
Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**GC/MS SEMIVOLATILES**  
**BY EPA METHOD 625 AND**  
**SW846 METHOD 8270C AND 8270D**  
**INCLUDING ADDITIONAL**  
**APPENDIX IX COMPOUNDS**

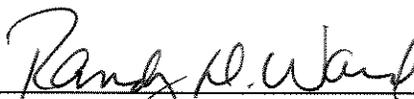
**SOP NUMBER:** **SOP-201**

**REVISION NUMBER:** **18**

**APPROVED BY:**



**SECTION MANAGER**



**QUALITY ASSURANCE OFFICER**

**EFFECTIVE DATE:** **09/16/08**

**DATE OF LAST REVIEW** **09/16/08**

## GC/MS SEMIVOLATILES

### BY EPA METHOD 625 AND SW846 METHOD 8270C and 8270D

#### 1.0 SCOPE AND APPLICATION

**Please see Appendix for definitions.**

This SOP (based primarily on SW-846 Method 8000B/8270C/8270D) is used for the analysis of semi-volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. A laboratory list of 625 and 8270 analytes with example limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8270C. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8270C/8270D, EPA 625 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or Technical Director.

#### 2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

#### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling. Soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed

#### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- 4.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.
- 4.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination.

## 5.0. EQUIPMENT AND APPARATUS

- 5.1 HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for splitless injection.
- 5.2 Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25  $\mu$ m film thickness fused silica capillary column or RTX-5 SIL-MS 30 m x 0.28 mm I.D. x 0.5 $\mu$ m film thickness.
- 5.3 HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.
- 5.4 HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.
- 5.5 HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.
- 5.6 Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- 5.7 Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.

## 6.0 REAGENTS

- 6.1 Methylene chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) – Trace analysis grade.
- 6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the BNA standards log book. The date they are opened is noted on the label and recorded in the BNA standards log book along with their lot number and vendor. Each standard that is prepared is recorded in the BNA standards log book and given a sequential number. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook. Makeup of common standards is detailed below. See the BNA standards log book for makeup of other standards.

- 6.2.1 The Decafluorotriphenylphosphine (DFTPP) tuning standard is prepared as follows (includes benzidine, pentachlorophenol and 4,4'-DDT): Using a 100 $\mu$ L syringe, 100 $\mu$ L (GCM-150, Ultra Scientific @ 1000 $\mu$ g/mL, or equivalent) is injected into a 2.0mL volumetric flask containing approximately 1.2mL methylene chloride (Trace Grade) and diluted to volume with same making a 50 $\mu$ g/mL standard. After capping and inverting several times, the solution is transferred into 2 labeled 2ml, teflon-lined, screw-capped vials and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months. A direct injection of 1.0 $\mu$ L is used to tune the instrument.
- 6.2.2 Calibration standards are prepared from a 200 $\mu$ g/mL working standard at a minimum of five concentrations. Calibration standards are prepared semi-annually unless the initial calibration verification standard indicates a problem. To makeup the 200 $\mu$ g/mL working standard inject the indicated amount of the following standards (or equivalent) into a 10mL volumetric containing approximately 5mL methylene chloride (Trace Grade) and dilute to volume with the same. After capping and inverting several times, the solution is transferred into an appropriate labeled vial, teflon-lined, screw-capped vial and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months. See the Appendix for analytes contained in these mixes.

<u>Standard (Interm.A in 10mls)</u>	<u>Conc (<math>\mu</math>g/mL)</u>	<u>Amount(<math>\mu</math>L)</u>	<u>Final (ug/mL)</u>
Base/Neutrals Mix 1 (Vendor, Lot#)	2000	1000	200
Benzidines Mix 2 (Vendor, Lot#)	2000	1000	200
Acids Mix (Vendor, Lot#)	2000	1000	200
SV Mix w/ Pyridine & Carbazole (Vendor, Lot#)	2000	1000	200
BN Surrogate (Vendor, Lot#)	5000	400	200
Acid Surrogate (Vendor, Lot#)	10,000	400	400
Catechol (Standard#)	10,000	200	200
Biphenyl (Standard#)	10,000	200	200
Caprolactum (Standard#)	10,000	200	200
Acetophenone (Standard#)	10,000	200	200

<u>Standard (Interm.B in 5mls)</u>	<u>Conc (<math>\mu</math>g/mL)</u>	<u>Amount(<math>\mu</math>L)</u>	<u>Final (ug/mL)</u>
Benzidine (Mix # 2)	2000	500	200

Standard (Interm.C in 5mls)

Atrazine (Standard#)	10,310	97	200
Benzaldehyde (Standard#)	10,590	94	200

**Additional Appendix IX compounds can be added using 3 additional mixes, AppIX A,B,C. Refer to standards log.**

To makeup the calibration standards, using a 1ml syringe add the appropriate amount of methylene chloride (trace grade) to a 2ml vial. Add the indicated amount of each intermediate standard to the vial. Add 20ul of internal standard to each screw-capped vial and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months.

<u>Amt. of 200ppm std</u>		<u>Amount of 2000ppm Internal Std</u>
<u>IntmA, Bor C</u>		
2 $\mu$ g/mL	10 $\mu$ L	20 $\mu$ L
5 $\mu$ g/mL	25 $\mu$ L	20 $\mu$ L
10 $\mu$ g/mL	50 $\mu$ L	20 $\mu$ L
20 $\mu$ g/mL	100 $\mu$ L	20 $\mu$ L
30 $\mu$ g/mL	150 $\mu$ L	20 $\mu$ L
40 $\mu$ g/mL	200 $\mu$ L	20 $\mu$ L
50 $\mu$ g/mL	250 $\mu$ L	20 $\mu$ L

60 µg/mL	300 µL	20 µL
70 µg/mL	350 µL	20 µL
80 µg/mL	400 µL	20 µL
90 µg/mL	450 µL	20 µL
100 µg/mL	500 µL	20 µL

The makeup of the 50µg/mL CCV standard is detailed below. Occasionally, unusual compounds are added to the mix so it is best to check the BNA standards log book for exact standard makeup. Note: MS list spikes and full list spikes for LCS and/or MS/MSD are prepared from an alternate source or lot number other than the calibration standards.

**Example: 50µg/mL CCV standard preparation:**

**SV3640**

using 1ml syringe add the appropriate amount of methylene chloride to a 2ml vial. Add the indicated amount of intermediate / stock standard to the vial. Add 20ul of internal standard to the vial (**SV 3635**). Soutlion stored in freezer at -15deg C+/-5degC for upto 1 week.

**50 ug/mL CCV**

	Stock Standard	Concentration		50 ug/mL	
<b>SV3515</b>	BNA intermediate mix	200 PPM		250 uL	
<b>SV3517</b>	Benzidine Int.	200 PPM		250 uL	
<b>SV3516</b>	Atrazine benzaldehyde Int.	200 PPM		250 uL	

6.2.3 The Initial Calibration Verification (ICV) standard is prepared from a vendor stock standard at a concentration of 200µg/mL as detailed below.

**Example: 50µg/mL ICV standard preparation:**

**(SV2100) ~ 50ug/ml ICV**

Into a 1 mL volumetric flask containing Methylene Chloride ~ about 0.6ml, add 250uls of **SV1995 (readystock 200ppm)** and dilute to volume.

Top off w/20uls of Internal Standard (**SV2079**)  
 Prep'd: 01/08/03                      Exp: 03/16/03

**(SV2396) ~ 50ug/ml ICV (benzaldehyde & atrazine)**

Into a 1 mL volumetric flask containing Methylene Chloride ~ about 0.6ml, add 250uls of **SV2305 (intermediate stock 200ppm)** and dilute to volume.

Top off w/20uls of Internal Standard (**SV2393**)  
 Prep'd: 09/18/03                      Exp: 03/18/04

**7.0 PROCEDURE**

Prior to using Federal Register 625, SW-846 8270C/8270D, or CLP (semivolatile method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3540, 3550, 3580, EPA method 625 or CLP).

7.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.

7.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (every 24 hours for *Federal Register* Method 625 except for South Carolina which only allows 12 hours). The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port and must meet the following criteria. Degradation of DDT to DDE and DDE should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible for 8270C. **Tailing factor should be 2.0 for 8270D for benzidine and pentachlorophenol.** For NPDES samples, the benzidine (base/neutral) tailing factor must not exceed 3.0 while the pentachlorophenol (acid) tailing factor cannot exceed 5.0. The calculation for tailing factors is best illustrated in Figure 13 of the *Federal Register* Method 625 which has been placed in the appendix. If degradation is excessive and/or poor chromatography is seen, the injection port may require cleaning and maintenance. It may also be necessary to break off 15-30cm of the capillary column. The mass spectrum of DFTPP is acquired as follows: by using one scan at the apex peak, or by using the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or using the average across the entire peak. Background subtraction is accomplished using a single scan or more than 20 scans prior to the elution of DFTPP.

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

**8270D has different tuning criteria for meeting DFTPP. See page 44 Table 3 of Method 8270D for criteria.**

### 7.3 Calibration

7.3.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed (only three concentration levels are required for *Federal Register* Method 625) and shown to meet the initial calibration criteria before any sample analysis may be performed. Method 625 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation

coefficient ( $r$ ) of  $\geq 0.995$  linear, 0.99 and six points for quadratic. Method 8270C requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as  $r$  is  $\geq 0.995$  linear, 0.99 and six points for quadratic. In addition, there are calibration check compounds (CCCs) which must have a %RSD less than 30% and system performance check compounds (SPCCs) which must meet a minimum average response factor of 0.050. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve are 2  $\mu\text{g/mL}$ , 5  $\mu\text{g/mL}$ , 10  $\mu\text{g/mL}$ , 20  $\mu\text{g/mL}$ , 30  $\mu\text{g/mL}$ , 40  $\mu\text{g/mL}$ , 50  $\mu\text{g/mL}$ , 60  $\mu\text{g/mL}$ , 70  $\mu\text{g/mL}$ , 80  $\mu\text{g/mL}$ , 90  $\mu\text{g/mL}$  and 100  $\mu\text{g/mL}$ . Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason and dated with the quantitation report and chromatogram. Refer to SOP-224 for guidance. All integrations are checked for acceptability. Response factors of less than 0.050 must be supported by the mass spectrum of the lowest standard. Retention times are set using the midpoint of the curve. **No quadratic curves are used for South Carolina. For 8270D the RSD for each target analyte should be less than or equal to 20% and each calibration level should meet the minimum response factors listed in Table 4. If the 20% RSD is not met, then the minimum correlation coefficient for the curve must be 0.99. If more than 10% of the compounds do not meet the 20% RSD or minimum correlation coefficient of 0.99, then the chromatographic system is considered too reactive to begin analysis. Injector maintenance should be performed and repeat the calibration procedure.**

CCCs:	<u>Base/Neutral</u>	<u>Acid</u>
	Acenaphthene	4-Chloro-3-methylphenol
	1,4-Dichlorobenzene	2,4-Dichlorophenol
	Hexachlorobutadiene	2-Nitrophenol
	N-Nitroso-di-phenylamine	Phenol
	Di-n-octyl-phthalate	Pentachlorophenol
	Fluoranthene	2,4,6-Trichlorophenol
	Benzo(a)pyrene	
SPCCs:	<u>Base/Neutral</u>	<u>Acid</u>
	N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
	Hexachlorocyclopentadiene	4-Nitrophenol

- 7.3.2 Initial Calibration Verification (ICV) - A second source standard at the 50  $\mu\text{g/mL}$  level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125% (**70-130% for 8270D**). If the second source recovery is above 125% or 130% for 8270D, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 75% or 70% for 8270D, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. If any compound in the ICV exceeds the criteria above, it may be evaluated and initialed by the organic section manager. If deemed acceptable,

the analyst may continue analysis. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. For ICV standard preparation refer to standard log book.

- 7.4 Continuing Calibration Verification (CCV)- Every 12 hours a CCV at 50 µg/mL must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for 8270C consists of the same SPCC requirements as the initial calibration. The CCCs must be less than or equal to 20% difference or drift (%D, calculations follow in section 7.9). If any of the CCCs do not meet the above limits, then all required analytes must be <20%D. Internal standard areas should be within 50 to 200 percent of the area of the curve midpoint or the previous CCV. Retention times for the internal standards should be within 30 seconds of the retention time of the curve midpoint or the previous CCV. Method 625 requires a %D of less than 20% for all required analytes. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30%D, it must be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis. **For 8270D, the 20% difference criteria must be applied to all compounds. If the criterion is not met for more than 20% of the compounds included in the initial calibration, then the GC system should undergo maintenance. If this does not solve the problem, then the initial calibration should be repeated. Each of the most common target analytes should meet the minimum response factors listed in Table 4. In situations where the failed compound is present, the data must be flagged as estimated. If the compound fails high in the CCV and is not present, the result can be reported as non-detect.**
- 7.5 LCS - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS containing all regular full list calibrated compounds is spiked into deionized water or sodium sulfate for soil using an alternate source or lot number than the calibration standards. See the LCS report forms in the appendix for example laboratory generated limits and the NPDES limits for 625 samples. Recoveries for the MS/MSD spike analytes in LCSs are charted annually to generate control limits for samples analyzed by method 8270C. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. If enough data points are not present to generate limits, the limits default to CLP spike limits for spike analytes or 10-100% for all other analytes. See Section 8.3 below for corrective action.  
**When analyzing samples for DOD QSM Version 3, DOD limits will be used.**
- 7.6 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch up to 20 samples. See Section 8.4 below for criteria and corrective action.
- 7.7 Samples - Prior to analysis, 1.0 mL samples are prepared by verifying volume and spiking with 20uL of the internal standard solution.

7.8 Instrument sequence-The instrument sequence log is filled out prior to sample analyses.

**An example of a typical instrument sequence log follows:**

- 1-DFTPP Tune (12:00 am)
- 2-CCV
- 3-LCS
- 4-Method Blank (or sample)
- 5-Sample
- 6-Sample
- 7-Sample
- 8-Sample
- 9-Sample
- 10-Sample
- 11-Sample-MS
- 12-Sample-MSD
- 13-Sample
- 14-Sample
- 15-Sample
- 16-DFTPP (12:00pm - 12 hours since last DFTPP/CCV)
- 17-CCV
- 18-Sample
- 19-Sample
- 20-Sample

7.9 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 and documented using the Analyst Data Review Checklist (see Appendix). **See SOP-224 for guidance on manual integrations.**

7.9.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the curve midpoint. Retention time should be within 30 seconds of the retention time of the curve midpoint. If not, the sample and historical data should be evaluated to determine the cause of the problem. If matrix effect is confirmed by reextraction/reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity.

7.9.2 Surrogates – Control limits are determined annually by charting LCSs and method blanks. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. All of the three surrogates for each fraction must be within the control limits in order for the extraction batch to be in control. If a surrogate exceeds the limits, the reason for the malfunction must be determined and a corrective action report must be completed. The sample must be reanalyzed, reextracted or flagged for QC problems. *Federal Register*

Method 625 contains no criteria for surrogate recovery. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

Surrogate	Water	Soil/Sediment
Nitrobenzene-d5	30-110	30-110
2-Fluorobiphenyl	35-110	35-110
Terphenyl-d14	55-125	40-120
Phenol-d6	15-110	30-110
2-Fluorophenol	15-110	25-110
2,4,6-Tribromophenol	45-125	30-115

- 7.9.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.
- 7.9.4 The qualitative identification of compounds is based on retention time and a comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum from the NBS database (NBS75K.I). This database is used as it contains relatively uncontaminated mass spectra of each target compound which cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes. Characteristic ions from the reference mass spectrum library are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. In addition, the following criteria must be met. The RRT of the sample analyte must be within 0.06 RRT units of the RRT of the standard analyte. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of the same ions in the reference spectrum. Structural isomers that produce very similar mass spectra should be identified as individual isomers so long as their GC retention times differ substantially. A library search may be made for analytes not associated with the calibration for the purpose of tentative identification. NOTE: The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.
- 7.9.5 Quantitation - Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response factor is 15%(20% 8270D) or less, then the concentration may be determined using the average response factor ( $\overline{RF}$ ) from the initial calibration data. Otherwise, the concentration must be determined from equations based on internal standard calibration using either linear or non-linear calibration. Calculations follow in Section 7.10.

## 7.10 Calculations:

7.10.1 The RF is calculated as follows: 
$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

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.

$C_{is}$  = Concentration of the internal standard.

7.10.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within  $\pm 20\%$  for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place. **20% difference for 8270D.**

7.10.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to  $\mu\text{g/L}$ .]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

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.

$C_{is}$  = Concentration of the internal standard in the volume extracted in  $\mu\text{g/L}$ .

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

$V_i$  = Volume of the extract injected ( $\mu\text{L}$ ). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$  = Mean response factor from the initial calibration.

$V_s$  = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.

7.10.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to  $\mu\text{g}/\text{kg}$ .]

7.10.5

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where:  $A_s$ ,

$A_{is}$ ,  $C_{is}$ ,  $D$ , and  $\overline{RF}$  are the same as for aqueous samples, and

$W_s$  = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.

## 8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

- 8.1 Internals - All samples and QC are spiked with internal standards. See section 7.9.1 above for criteria and corrective action.
- 8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control control limits for the surrogates. See section 7.9.2 above for criteria and corrective action. If any surrogate recoveries are below 10%, samples must be re-extracted if sample is available.
- 8.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the data must be evaluated for flagging due to QC problems.
- 8.4 Method Blanks - The concentration of all method target analytes should be below the MDL for each method target analyte (<RL for common lab contaminants and <1/2 RL for other targets for DOD QSM Ver.3 projects). The first step of corrective

action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, may need to be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank.

- 8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the full list spiking solution is used). Control limits for the MS/MSD recoveries are the same as those for the LCS found in the appendix. RPD limits are found on the LCS report forms in the appendix. Samples which do not meet these criteria due to matrix must be evaluated for flagging on the final report due to QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

- 8.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
- 8.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 8.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.
- 8.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.
- 8.5.5 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

## 9.0 HEALTH AND SAFETY

- 9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
- 9.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 9.3 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

## 10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 10.1 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.
- 10.2 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 11.0 TABLE OF DEFINITIONS, REFERENCES & EXAMPLE FORMS

Definitions and examples of the LCS report sheets (625 water limits, in-house water limits and in-house soil limits), the analyst data review checklist and figure 13 from *Federal Register* method 625 for the tailing factor calculation are located in the appendix. **When analyzing samples for DOD QSM Version 3, DOD limits will be used for surrogates, LCS, and MS/MSD.**

## **APPENDIX**

## TABLE OF DEFINITIONS

1. amu- atomic mass unit
2. BNA- Base neutral/acid
3. °C- degrees Centigrade
4. CCC- Calibration Check Compound
5. CCV- Continuing Calibration Verification
6. CLP- Contract Laboratory Program
7. %D- percent difference
8. DFTPP- Difluorotriphenylphosphine
9. EICP- extracted ion current profile
10. EPA- Environmental Protection Agency
11. g- gram or grams
12. GC- Gas Chromatograph
13. GC/MS- Gas Chromatograph/Mass Spectrometer
14. ICV- Initial Calibration Verification
15. I.D.- inner diameter
16. ISTD- internal standard
17. LSC- Laboratory Sample Concentrator
18. MDL- method detection limit
19. MS- Matrix Spike
20. MSD- Matrix Spike Duplicate
21. M.S.- Mass Spectrometer
22. µm- micrometer
23. µL- microliter
24. mL- milliliter
25. mm- millimeter
26. ng- nanogram
27. NPDES- National Pollutant Discharge Elimination System
28. P&T- purge and trap
29. QC- quality control
30. %R- percent recovery
31. RPD- relative percent difference
32. RRT- relative retention time
33. %RSD- percent relative standard deviation
34. SOP- Standard Operating Procedure
35. Surr.- surrogate
36. SPCC- System Performance Check Compound
37. TCLP- Toxicity Characteristic Leaching Procedure
38. USACE- United States Army Corps Of Engineers
39. LCS – Lab Control Sample

**Refer to SOP-431 for additional definitions**

## REFERENCES

1. *40 CFR, Part 136; Appendix A*
2. *Test Methods for Evaluating Solid Waste, SW-846*
3. *National Environmental Laboratory Accreditation Conference; CH. 5, 2003*
4. *USACE, EM 200-1-3; Appendix 1; Shell, 2/2001*
5. *DOD Quality Systems Manual for Environmental Laboratories, Ver.3, 01/2006*

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (625)

Lab Name: Empirical Laboratories      Contract:  
 Lab Code: NA      Batch No.: NA      SAS No.: NA      SDG No.:  
 Matrix Spike    -    Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

COMMENTS: \_\_\_\_\_

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (625)

Lab Name: Empirical Laboratories      Contract:  
 Lab Code: NA      Batch No.: NA      SAS No.: NA      SDG No.:  
 Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

RPD: 0 out of 0 outside limits  
 Spike Recovery: 0 out of 56 outside limits

COMMENTS: \_\_\_\_\_

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (In-House)

Lab Name: Empirical Laboratories Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.0	0.0000	100.00	100	40-112
4-Chloro-3-methylphenol	200.0	0.0000	200.00	100	32-110
2-Chlorophenol	200.0	0.0000	200.00	100	29-110
1,4-Dichlorobenzene	100.0	0.0000	100.00	100	26-110
2,4-Dinitrotoluene	100.0	0.0000	100.00	100	46-122
4-Nitrophenol	200.0	0.0000	200.00	100	21-110
N-Nitroso-di-n-prop.(1)	100.0	0.0000	100.00	100	39-110
Pentachlorophenol	200.0	0.0000	200.00	100	32-133
Phenol	200.0	0.0000	200.00	100	10-110
Pyrene	100.0	0.0000	100.00	100	51-137
1,2,4-Trichlorobenzene	100.0	0.0000	100.00	100	28-110

COMPOUND	SPIKE ADDED (UG/L)	MSD CONCENTRATION (UG/L)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
Acenaphthene	100.0	100.00	100	0	31	40-112
4-Chloro-3-methylphenol	200.0	200.00	100	0	42	32-110
2-Chlorophenol	200.0	200.00	100	0	40	29-110
1,4-Dichlorobenzene	100.0	100.00	100	0	28	31-110
2,4-Dinitrotoluene	100.0	100.00	100	0	38	46-122
4-Nitrophenol	200.0	200.00	100	0	50	21-110
N-Nitroso-di-n-prop.(1)	100.0	100.00	100	0	38	39-110
Pentachlorophenol	200.0	200.00	100	0	50	32-133
Phenol	200.0	200.00	100	0	42	10-110
Pyrene	100.0	100.00	100	0	31	51-137
1,2,4-Trichlorobenzene	100.0	100.00	100	0	28	28-110

(1) N-Nitroso-di-n-propylamine  
 # Column to be used to flag recovery and RPD values with an asterisk  
 \* Values outside of QC limits

RPD: 0 out of 11 outside limits  
 Spike Recovery: 0 out of 22 outside limits

COMMENTS: \_\_\_\_\_

FORM 3

SOIL SEMIVOLATILE LAB CONTROL SAMPLE (In-House)

Lab Name: Empirical Laboratories Contract:  
 Lab Code: ELABN Case No.: NA SAS No.: NA SDG No.:  
 Matrix Spike - Client Sample No.: Level:(low/med) LOW

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	10000	0.0000	10000	100	27-116
4-Chloro-3-methylphenol	20000	0.0000	20000	100	18-111
2-Chlorophenol	20000	0.0000	20000	100	10-110
1,4-Dichlorobenzene	10000	0.0000	10000	100	14-110
2,4-Dinitrotoluene	10000	0.0000	10000	100	34-110
4-Nitrophenol	20000	0.0000	20000	100	24-120
N-Nitroso-di-n-prop.(1)	10000	0.0000	10000	100	24-112
Pentachlorophenol	20000	0.0000	20000	100	10-114
Phenol	20000	0.0000	20000	100	10-110
Pyrene	10000	0.0000	10000	100	33-150
1,2,4-Trichlorobenzene	10000	0.0000	10000	100	19-110

COMPOUND	SPIKE ADDED (ug/Kg)	LCSD CONCENTRATION (ug/Kg)	LCSD % REC #	% RPD #	QC LIMITS RPD	REC.
Acenaphthene	10000	10000	100	0	19	27-116
4-Chloro-3-methylphenol	20000	20000	100	0	42	18-111
2-Chlorophenol	20000	20000	100	0	50	10-110
1,4-Dichlorobenzene	10000	10000	100	0	27	14-110
2,4-Dinitrotoluene	10000	10000	100	0	47	34-110
4-Nitrophenol	20000	20000	100	0	50	24-120
N-Nitroso-di-n-prop.(1)	10000	10000	100	0	38	24-112
Pentachlorophenol	20000	20000	100	0	47	40-110
Phenol	20000	20000	100	0	35	10-110
Pyrene	10000	10000	100	0	36	33-150
1,2,4-Trichlorobenzene	10000	10000	100	0	23	19-110

(1) N-Nitroso-di-n-propylamine  
 # Column to be used to flag recovery and RPD values with an asterisk  
 \* Values outside of QC limits  
 RPD: 0 out of 11 outside limits  
 Spike Recovery: 0 out of 22 outside limits

COMMENTS: \_\_\_\_\_

**BNA STANDARDS USED**

<u>base/neutral mix (2000ppm)</u>	<u>acids mix (2000ppm)</u>
bis(2-Chloroethyl)ether	2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether	2-Methylphenol
1,3-Dichlorobenzene	4-Methylphenol
1,2-Dichlorobenzene	Benzoic acid
1,4-Dichlorobenzene	4,6-Dinitro-2-methylphenol
Hexachloroethane	4-Nitrophenol
N-Nitroso-di-methylamine	2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine	2,4,6-Trichlorophenol
2,4-Dinitrotoluene	Phenol
2,6-Dinitrotoluene	Pentachlorophenol
Fluorene	2-Nitrophenol
Dimethylphthalate	4-Chloro-3-methylphenol
Hexachlorocyclopentadiene	2,4-Dichlorophenol
Anthracene	2,4-Dimethylphenol
4-Bromophenyl-phenylether	Benzoic acid
Di-n-butylphthalate	
bis(2-Chloroethoxy)methane	
1,2-Diphenylhydrazine	<u>semivoa misc.mix(2000ppm)</u>
Fluoranthene	Aniline
Hexachlorobenzene	Benzyl alcohol
N-Nitrosodiphenylamine	Carbazole
Phenanthrene	4-Chloroaniline
Hexachlorobutadiene	Dibenzofuran
Isophorone	2-Methylnaphthalene
Naphthalene	2-Nitroaniline
Nitrobenzene	3-Nitroaniline
1,2,4-Trichlorobenzene	4-Nitroaniline
Acenaphthene	Pyridine
Acenaphthylene	
2-Chloronaphthalene	<u>Benzidine mix (2000ppm)</u>
4-Chlorophenyl-phenylether	Benzidine
Diethylphthalate	3,3'-Dichlorobenzidine
Benzo(a)anthracene	
Bis(2-ethylhexyl)phthalate	
Butylbenzylphthalate	<u>individual mixes (10,000ppm)</u>
Chrysene	Caprolactam
p-(Dimethylamino)azobenzene	Benzaldehyde
Pyrene	Atrazine
Benzo(b)fluoranthene	1,1'-Biphenyl
Benzo(k)fluoranthene	Catechol
Benzo(g,h,i)perylene	
Benzo(a)pyrene	
Dibenz(a,h)anthracene	
Di-n-octylphthalate	
Indeno(1,2,3-cd)pyrene	

INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE					
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 (I.S) (79)		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 (I.S)(35)		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 (I.S) (92)		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 (I.S) (59)		164	Perylene-d12 (I.S) (101)		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278

Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276
<b>I.S=internal standard, Q.M=quant mass, S=surrogate</b>					

<b>INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE</b>					
<b>COMPOUND</b>	<b>*I.S</b>	<b>Q.M</b>	<b>COMPOUND</b>	<b>*I.S</b>	<b>Q.M</b>
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolidine	1	100		59	
N-Nitrosomorpholine	1	56		59	
O-Toluidine	1	106		59	
	1			59	
	1			59	
	1			59	
	1		Phenanthrene-d10 (I.S) (79)		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 (I.S)(35)		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 (I.S) (92)		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 (I.S) (59)		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 (I.S) (101)	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	

I.S.=internal standard, Q.M=quant mass, S=surrogate
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**Internal Standards & Surrogates**

<b>BNA internals (2000ppm)</b>		<b>Acid surrogate (7500ppm)</b>
1,4-Dichlorobenzene-d4 (I.S)(1)		2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)		Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)		2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)		<b>2,-Chlorophenol-d4 (S)</b>
Chrysene-d12 (I.S) (92)		
Perylene-d12 (I.S) (101)		<b>BN surrogate (5000ppm)</b>
		Nitrobenzene-d5 (S)
		Terphenyl-d14 (S)
		2-Fluorobiphenyl (S)
		<b>1,2-Dichlorobenzene-d4 (S)</b>

**Reporting Limits**

	Soil ug/KG	Water ug/L		Soil ug/KG	Water ug/L
Pyridine	330	5.0	Acenaphthylene	330	5.0
N-Nitroso-di-methylamine	330	5.0	2,6-Dinitrotoluene	330	5.0
Aniline	330	5.0	3-Nitroaniline	1300	20
Phenol	330	5.0	Acenaphthene	330	5.0
bis(2-Chloroethyl)ether	330	5.0	2,4-Dinitrophenol	3300	50
2-Chlorophenol	330	5.0	4-Nitrophenol	1300	20
1,3-Dichlorobenzene	330	5.0	Dibenzofuran	330	5.0
1,4-Dichlorobenzene	330	5.0	2,4-Dinitrotoluene	330	5.0
Benzyl alcohol	330	5.0	Diethylphthalate	330	5.0
1,2-Dichlorobenzene	330	5.0	4-Chlorophenyl-phenylether	330	5.0
2-Methylphenol	330	5.0	Fluorene	330	5.0
bis(2-Chloroisopropyl)ether	330	5.0	4-Nitroaniline	1300	20
3-Methylphenol	330	5.0	2,3,4,6-Tetrachlorophenol	330	5.0
4-Methylphenol	330	5.0	1,1'-Biphenyl	330	5.0
N-Nitroso-di-n-propylamine	330	5.0	1,2-Diphenylhydrazine	330	5.0
Hexachloroethane	330	5.0	Carbazole	330	5.0
Benzaldehyde	330	5.0	4,6-Dinitro-2-methyl phenol	1300	20
Nitrobenzene	330	5.0	N-Nitrosodiphenylamine	330	5.0
Isophorone	330	5.0	4-Bromophenyl-phenylether	330	5.0
2-Nitrophenol	330	5.0	Hexachlorobenzene	330	5.0
2,4-Dimethylphenol	1300	20	Pentachlorophenol	1300	20
Benzoic acid	3300	50	Phenanthrene	330	5.0
bis(2-Chloroethoxy)methane	330	5.0	Anthracene	330	5.0
2,4-Dichlorophenol	330	5.0	Di-n-butylphthalate	330	5.0
1,2,4-Trichlorobenzene	330	5.0	Fluoranthene	330	5.0
Naphthalene	330	5.0	Atrazine	330	5.0
4-Chloroaniline	330	5.0	Benzidine	3300	50
Hexachlorobutadiene	330	5.0	Pyrene	330	5.0
4-Chloro-3-methylphenol	330	5.0	Butylbenzylphthalate	330	5.0
2-Methylnaphthalene	330	5.0	3,3'-Dichlorobenzidine	330	5.0
1-Methylnaphthalene	330	5.0	Benzo(a)anthracene	330	5.0
Acetophenone	330	5.0	Chrysene	330	5.0
1,2,4,5-Tetrachlorobenzene	330	5.0	Bis(2-ethylhexyl)phthalate	330	5.0
Caprolactam	330	5.0	Di-n-octylphthalate	330	5.0
Hexachlorocyclopentadiene	330	5.0	Benzo(b)fluoranthene	330	5.0
2,4,6-Trichlorophenol	330	5.0	Benzo(k)fluoranthene	330	5.0
2,4,5-Trichlorophenol	330	5.0	Benzo(a)pyrene	330	5.0
2-Chloronaphthalene	330	5.0	Indeno(1,2,3-cd)pyrene	330	5.0
2-Nitroaniline	1300	20	Dibenz(a,h)anthracene	330	5.0
Dimethylphthalate	330	5.0	Benzo(g,h,i)perylene	330	5.0

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>	
<b>Batch Number(s):</b>	
<b>Method:</b>	<b>8260B/8270C/8270D (Circle One)</b>

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?	_____	_____	_____	_____ _____ _____
2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.( eg. m/p-xylene, ketones,etc.).	_____	_____	_____	_____ _____ _____
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?	_____	_____	_____	_____ _____ _____
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs.	_____	_____	_____	_____ _____ _____
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?	_____	_____	_____	_____ _____ _____
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?	_____	_____	_____	_____ _____ _____
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?	_____	_____	_____	_____ _____ _____
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?	_____	_____	_____	_____ _____ _____

Comments on any "No" response:

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Primary-Level Review: \_\_\_\_\_

Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**GC/MS VOLATILES**

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**BY EPA METHOD 624 AND**

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**SW-846 METHOD 8260B**

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**INCLUDING APPENDIX IX  
COMPOUNDS**

**SOP NUMBER:**

**SOP-202**

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**REVISION NUMBER:**

**21**

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**APPROVED BY:**



**SECTION MANAGER**

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**QUALITY ASSURANCE OFFICER**

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**EFFECTIVE DATE:**

**09/11/08**

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**DATE OF LAST REVIEW**

**09/11/08**

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**GC/MS VOLATILES  
BY EPA METHOD 624 AND SW-846 METHOD 8260B**

## **1.0 SCOPE AND APPLICATION**

**Please see Appendix for definitions.**

This SOP (based primarily on SW-846 Method 8260B) is used for the analysis of volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. The normal laboratory list of analytes with their typical LCS limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8260B. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8260B, EPA 624 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or the Technical Director.

## **2.0 METHOD SUMMARY**

After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

## **3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE**

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. All water and non-quarantined soil volatile samples are stored in the Hobart in the sample storage room at a temperature of 4°C. Quarantined soil volatile samples are stored in the dorm-size refrigerator in the sample receiving/log-in room at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).

## **4.0 INTERFERENCES AND POTENTIAL PROBLEMS**

Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

## 5.0. EQUIPMENT AND APPARATUS

- 5.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.
- 5.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4  $\mu$ m film thickness or 20 meter x 0.18 mm ID 1.0  $\mu$ m film thickness silicon coated fused silica capillary column or equivalent.
- 5.3 M.S. : HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1  $\mu$ L (50 ng) of the GC/MS tuning standard are injected through the GC.]
- 5.4 Purge and Trap Unit
  - 5.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.
  - 5.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.
- 5.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- 5.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.
- 5.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000  $\mu$ L.
- 5.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.
- 5.9 Balance - analytical, 0.0001 g; top-loading, 0.1 g.
- 5.10 Disposable pasteur pipets.
- 5.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.
- 5.12 Spatula - stainless steel.
- 5.13 Glass scintillation vials - 20mL with screw caps.
- 5.14 Nitrile Gloves
- 5.15 pH paper (measures pH from 0-14).

**6.0 REAGENTS AND SOLUTIONS**

- 6.1 Organic-free reagent water - obtained from a modulab system.
- 6.2 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)
- 6.3 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484-1, or equivalent)
- 6.4 Sodium bisulfate, NaHSO<sub>4</sub> – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- 6.5 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the VOA standards and reagents logbook along with their lot number and vendor. Each standard that is prepared is recorded in the VOA standards and reagents logbook and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date.** All stocks and standards are stored in the freezer at a temperature of  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  or less from the date they are received/prepared. The freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See the VOA standards log book for makeup of other standards.
- 6.5.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50 $\mu\text{L}$  syringe, 40 $\mu\text{L}$  of standard (BFB @ 2500ng/ $\mu\text{L}$ ) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same making a 50ng/ $\mu\text{L}$  standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  or less for up to 6 months(1 week for South Carolina samples). A direct injection of 1 $\mu\text{L}$  is used to tune the instrument.
- 6.5.2 The internal and surrogate standard is prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor, Lot) and diluted to volume with same making a 150ng/ $\mu\text{L}$  standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1 $\mu\text{L}$  of this standard. The internal standard/surrogate solution will be replaced if the -50%-200% criteria fails in the CCV.

Standard	Conc. (ng/ $\mu\text{L}$ )	Syringe ( $\mu\text{L}$ )	Amount ( $\mu\text{L}$ )
8260 ISTD/Surr. Mix (Vendor, Lot)	2500	1000	3000

- 6.5.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual

compounds are added to the mix so it is best to check the VOA standards log book for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard. See the appendix for analytes in the main mixes.

6.5.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50μg/L (5mL purge) standard is made using 25μL of this standard to 50mL of reagent water.

Stock Standard(CCV)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE (Cat#30265) (Vendor, Lot)	20000	25	20	200
Vinyl Acetate (#3766) (Vendor, Lot)	5000	100	80	200
Ketones (cat#30006) (Vendor, Lot)	5000	100	80	200
Liquid mix (C-349H-07) (Vendor, Lot)	2000	100	100	100
Custom mix (CCS-1037) (Vendor, Lot)	5000	50	40	100
Gases (cat#30042) (Vendor, Lot)	2000	100	100	100
Acrolein/Acrylonitrile (CC2098.10) (Vendor, Lot)	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standards log.

6.5.4 ICV/LCS/MATRIX SPIKE MIX: A second source standard is to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50μg/L (5mL purge) ICV/LCS/Matrix Spike is made using 25μL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE(Vendor, Lot#)	20,000	25	20	200

Vinyl Acetate(Vendor,Lot#)	5000	100	80	200
Ketones(Vendor,Lot#)	5000	100	80	200
Liquid mix (Vendor,Lot#)	2000	100	100	100
Custom Mix(Vendor,Lot#)	5000	50	40	100
Gases (Vendor,Lot#)	2000	100	100	100
Acrolein/Acrylonitrile (Vendor,Lot#)	50,000	50	50	500

## 7.0 PROCEDURE

7.1 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

7.2 System Bakeout - Prior to analysis, the autosampler purge and trap unit is stepped to bake and an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

7.3 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0 $\mu$ L injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours). The mass spectrum of BFB is acquired as follows: by using one scan at the apex peak, or by using the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or using the average across the entire peak. Background subtraction is accomplished using a single scan no more than 20 scans prior to the elution of BFB.

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

7.4 Calibration: Calibration standards are made up in water using the appropriate amount of the methanol standard. Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate. All calibration standard integrations must be checked for acceptability.

7.4.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations. Method 624 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient ( $r$ ) of  $\geq 0.995$ . Method 8260B requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as  $r$  is  $\geq 0.995$  linear or  $\geq 0.99$  quadratic. In addition, there are calibration check compounds (CCCs) listed below which must have a %RSD less than 30% and five system performance check compounds (SPCCs) which must meet the average response factor criteria listed below. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride
SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

7.4.2 Initial Calibration Verification - A second source standard at the 50  $\mu\text{g/L}$  (5mL purge) level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. If the second source recovery is above 125%, the main standard has probably deteriorated for that compound. That standard must be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard has probably deteriorated for that compound and must be replaced. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram.

7.4.3 Continuing Calibration Verification (every 12 hours) - A midpoint calibration standard (generally 50  $\mu\text{g/L}$  - 5mL purge) must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for method 8260B consists of the same SPCC criteria as above and  $\leq 20\%$  drift or difference (calculations given in section 7.10) for the CCCs as listed above. The internal standards must also be evaluated as listed below. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing

calibration not subject to the criteria above exceeds 30% D, it must be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis.

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

#### Internal standard checks

7.4.3.1.1 Retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.3.1.2 Response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 LCS - An LCS is analyzed every 12 hour tune. Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See section 8.3 below for criteria and corrective action. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

7.6 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See section 8.4 below for criteria and corrective action.

7.7 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP-225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

7.7.1. Load the vial into the Archon autosampler in the expected position.

7.7.2. Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation

sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per matrix.

- 7.7.3. After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2. If it is not, record the pH and generate a corrective action report. The sample report will have to be flagged for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

#### 7.8 Instrument sequence

**An example of a typical instrument sequence log follows:**

1-BFB Tune (12:00 am)  
2-CCV  
3-LCS  
4-Method Blank  
5-Sample  
6-Sample  
7-Sample  
8-Sample  
9-Sample  
10-Sample  
11-Sample  
12-Sample  
13-Sample  
14-Sample  
15-Sample  
16-Sample  
17-Sample MS  
18-Sample MSD  
19-BFB (12:00pm - 12 hours since last BFB/CCV)  
20-CCV  
21-LCS  
22-Method Blank  
23-Sample  
24-Sample

- 7.9 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 7.10 below. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 (documented using the USACE Analyst Data Review Checklist for USACE projects). **See SOP-224 for guidance on manual integrations.**

- 7.9.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the continuing calibration. Retention time should be within 30 seconds of the retention time of the continuing calibration. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample

analysis validity. If not, the sample and historical data should be evaluated to determine the cause of the problem. Reanalysis is expected if it appears to be from a leak. If matrix effect is confirmed by reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect.

- 7.9.2 Surrogates – Control limits are determined by charting LCSs and method blanks. All of the surrogates must be within these limits in order for the analysis to be in control. If not, the reason for the malfunction must be determined and reanalysis may be necessary. If historical data indicates matrix, the sample would be flagged appropriately. When the surrogates exceed either the control limits, a corrective action report must be completed. *Federal Register* Method 624 contains no criteria for surrogate recovery. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

Surrogate	WATER	SOIL/SEDIMENT
Dibromofluoromethane	85-120	80-120
1,2-Dichloroethane-d4	85-130	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

- 7.9.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed, the next sample must be checked for carryover and the sparge position must be checked for contamination through the analysis of a system blank. Any dilution should keep the concentration of the analyte in question within the mid-range of the curve.
- 7.9.4 Qualitative identification is made as indicated below.
- 7.9.4.1 The mass spectra are compared to reference spectra in a user-created data base especially compiled to contain relatively uncontaminated mass spectra of each target compound. Note: Such a file cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes.
- 7.9.4.2 The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

#### 7.10 Calculations:

7.10.1 The RF is calculated as follows: 
$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A<sub>s</sub> = Peak area (or height) of the analyte or surrogate.

A<sub>is</sub> = Peak area (or height) of the internal standard.

C<sub>s</sub> = Concentration of the analyte or surrogate.

C<sub>is</sub> = Concentration of the internal standard.

7.10.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

7.10.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

where:

A<sub>s</sub> = Area (or height) of the peak for the analyte in the sample.

A<sub>is</sub> = Area (or height) of the peak for the internal standard.

C<sub>is</sub> = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V<sub>i</sub> = For purge-and-trap analysis, V<sub>i</sub> is not applicable and is set at 1.

$\overline{RF}$  = Mean response factor from the initial calibration.  
 $V_s$  = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

7.10.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(W_s)(1000)}$$

where:  $A_s$ ,

$A_{is}$ ,  $C_{is}$ ,  $D$ , and  $\overline{RF}$  are the same as for aqueous samples, and  
 $W_s$  = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

## 8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

- 8.1 Internal Standards - All samples and QC are spiked with internals. See section 7.9.1 above for criteria and corrective action.
- 8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits. See section 7.9.2 above for criteria and corrective action.
- 8.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20  $\mu\text{g/L}$  when analyzing 624 samples (QC Check Sample). The recoveries are used to generate control limits. See the LCS report forms in the appendix for the laboratory generated limits. The limits are in-house generated matrix spike limits or client specified limits for matrix spike analytes and 70-130% (or client specified limits) recovery for waters or soils for all other analytes if limits have not been generated. Limits for 624 LCSs are taken from table 5 of method 624. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reanalyzed for the failed analytes only. **When analyzing samples for DOD QSM Version 3, DOD limits will be used. South Carolina limits are 70-130% except for poor purgers which are 60-140%.**
- 8.4 Method Blanks - The concentration for most method target analytes must be  $< \frac{1}{2}$  the reporting limit. Common laboratory contaminants, such as acetone and methylene chloride, must be  $<$  the reporting limit. The first step of corrective action is to assess the affect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this

contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. A method blank is analyzed every 12 hour tune.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. Criteria for the MS/MSD recoveries are the same as the LCS limits. Limits for the RPDs are 30% RPD for water and soil.. Samples that do not meet these criteria due to matrix must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

8.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

8.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

8.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

8.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

8.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

<u>CHROMATOGRAPHIC COND.</u>	<u>VOA1</u>	<u>VOA3</u>	<u>VOA4</u>	<u>VOA5</u>
<u>GC</u>	<u>5890 SERIES II</u>	<u>6890</u>	<u>5890 SERIES II</u>	<u>5890 SERIES II</u>
<u>MS</u>	<u>5971</u>	<u>5973</u>	<u>5972</u>	<u>5972</u>
GC COLUMN	RTX-VRX- 60mX0.25mmX1.4um	RTX-VRX- 60mX0.25mmX1.4um	DBVRX- 60mX0.25mmX1.4um	RTX-VRX-60mX0.25mmX1.4um
INJECTOR LINER	1.0MM	1.0MM	2.0MM	2.0MM
INJECTOR				
TEMPERATURE	150deg C	150deg C	150deg C	150deg C
TRANSFER LINE TEMP.	250deg C	<b>150deg C</b>	250deg C	250deg C

**EMPIRICAL LABORATORIES, LLC**

SOP-202  
 Revision:21  
 Date: 09/11/08  
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COLUMN PRESSURE	9.0PSI	X	X	X
COLUMN FLOW	X	1.1 ml/min	1.1 ml/min	1.2 ml/min
SPLIT FLOW/RATIO	<a href="#">19ml/min@50deg C</a>	43.3ml/min (40:1)	<a href="#">20ml/min@50deg C</a>	<a href="#">20ml/min@50deg C</a>
INITIAL TEMPERATURE	<b>45deg C</b>	<b>50deg C</b>	<b>50deg C</b>	<b>50deg C</b>
INITIAL HOLD TIME	<b>5.0 min</b>	<b>10 min</b>	<b>10 min</b>	<b>10 min</b>
RAMP1	<b>9deg C / min</b>	<b>25deg C / min</b>	<b>25deg C / min</b>	<b>25deg C / min</b>
TEMPERATURE 1 / TIME 1	<b>240deg C/ 6.1 min</b>	<b>220deg C/ 6.0 min</b>	<b>220deg C/ 7.2 min</b>	<b>220deg C/ 6.0 min</b>
RAMP2	X	X	X	X
TEMPERATURE 2 / TIME 2	X	X	X	X
TOTAL RUN TIME	32.7 min	23.0 min	24.2 min	23.0 min
<b>CONCENTRATOR</b>	<b>(Tekmar 2000)</b>	<b>(Tekmar 3100)</b>	<b>(Tekmar 3000)</b>	<b>(Tekmar 3100)</b>
<b><u>ARCHON PURGE COND.</u></b>				
VALVE/LINE TEMP.	125deg / 125deg C	110deg / 110deg C	130deg / 130deg C	130deg / 130deg C
PURGE TIME	11.0 min	11.0 min	11.0 min	11.0 min
PURGE FLOW	45ml/min	40ml/min	40ml/min	40ml/min
DRY PURGE TIME	2.0 min	2.0 min	2.0 min	2.0 min
DESORB PREHEAT	245deg C	255deg C	255deg C	255deg C
DESORB TEMPERATURE	250deg C	260deg C	260deg C	260deg C
<b>DESORB TIME</b>	<b>2.0 min</b>	<b>1.0 min</b>	<b>1.0 min</b>	<b>1.0 min</b>
BAKE TEMPERATURE	270deg C	270deg C	270deg C	270deg C
BAKE TIME	9.0 min	10.0 min	10.0 min	10.0 min

**RTX columns are from RESTEK & DBVRX columns are from J&W.**

## 9.0 HEALTH AND SAFETY

- 9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- 9.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
- 9.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the office next to the technical director.

## 10. WASTE MANAGEMENT AND POLLUTION PREVENTION

- 10.1 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.
- 10.2 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 11.0 EXAMPLE FORMS

Definitions and examples of the instrument run log, LCS report sheets (624, water and soil) and the USACE analyst data review checklist are located in the following Appendix.

**When analyzing samples for DOD QSM Version 3, DOD limits will be used for surrogates, LCS, and MS/MSD.**

**APPENDIX****TABLE OF DEFINITIONS**

1. amu- atomic mass unit
2. BFB- Bromofluorobenzene
3. °C- degrees Centigrade
4. CCC- Calibration Check Compound
5. CCV- Continuing Calibration Verification
6. CLP- Contract Laboratory Program
7. %D- percent difference
8. EICP- extracted ion current profile
9. EPA- Environmental Protection Agency
10. g- gram or grams
11. GC- Gas Chromatograph
12. GC/MS- Gas Chromatograph/Mass Spectrometer
13. ICV- Initial Calibration Verification
14. I.D.- inner diameter
15. ISTD- internal standard
16. LSC- Laboratory Sample Concentrator
17. MDL- method detection limit
18. MS- Matrix Spike
19. MSD- Matrix Spike Duplicate
20. M.S.- Mass Spectrometer
21. µm- micrometer
22. µL- microliter
23. mL- milliliter
24. mm- millimeter
25. ng- nanogram
26. P&T- purge and trap
27. QC- quality control
28. %R- percent recovery
29. RPD- relative percent difference
30. %RSD- percent relative standard deviation
31. SOP- Standard Operating Procedure
32. Surr.- surrogate
33. SPCC- System Performance Check Compound
34. TCLP- Toxicity Characteristic Leaching Procedure
35. USACE- United States Army Corps Of Engineers
36. VOA- volatile organic analysis
37. LCS- Lab Control Sample

**REFERENCES**

1. *40 CFR, Part 136; Appendix A*
2. *Test Methods for Evaluating Solid Waste, SW-846, Third Edition*
3. *National Environmental Laboratory Accreditation Conference; CH. 5, 2001*
4. *USACE, EM 200-1-3; Appendix 1; Shell, 2/2001*
5. *DOD Quality Systems Manual for Environmental Laboratories, 6/2002*

FORM 3

WATER VOLATILE LAB CONTROL SAMPLE (624)

Lab Name: Empirical Laboratories, LLC Contract: ELAB

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acrolein	100.00	0.0000	100.000	100	NML
Acrylonitrile	100.00	0.0000	100.000	100	NML
Benzene	20.00	0.0000	20.000	100	64-136
Bromodichloromethane	20.00	0.0000	20.000	100	66-134
Bromoform	20.00	0.0000	20.000	100	71-129
Bromomethane	20.00	0.0000	20.000	100	14-186
Carbon tetrachloride	20.00	0.0000	20.000	100	73-127
Chlorobenzene	20.00	0.0000	20.000	100	66-134
Chloroethane	20.00	0.0000	20.000	100	38-162
2-Chloroethyl vinyl eth	40.00	0.0000	40.000	100	D-224
Chloroform	20.00	0.0000	20.000	100	68-132
Chloromethane	20.00	0.0000	20.000	100	D-204
Dibromochloromethane	20.00	0.0000	20.000	100	68-132
1,2-Dichlorobenzene	20.00	0.0000	20.000	100	63-137
1,3-Dichlorobenzene	20.00	0.0000	20.000	100	73-127
1,4-Dichlorobenzene	20.00	0.0000	20.000	100	63-137
1,1-Dichloroethane	20.00	0.0000	20.000	100	73-127
1,2-Dichloroethane	20.00	0.0000	20.000	100	68-132
1,1-Dichloroethene	20.00	0.0000	20.000	100	51-149
trans-1,2-Dichloroethen	20.00	0.0000	20.000	100	70-130
1,2-Dichloropropane	20.00	0.0000	20.000	100	34-166
cis-1,3-Dichloropropene	20.00	0.0000	20.000	100	24-176
trans-1,3-Dichloroprope	20.00	0.0000	20.000	100	50-150
Ethylbenzene	20.00	0.0000	20.000	100	59-141
Methylene chloride	20.00	0.0000	20.000	100	61-139
1,1,2,2-Tetrachloroetha	20.00	0.0000	20.000	100	61-139
Tetrachloroethene	20.00	0.0000	20.000	100	74-126
Toluene	20.00	0.0000	20.000	100	75-125
1,1,1-Trichloroethane	20.00	0.0000	20.000	100	75-125
1,1,2-Trichloroethane	20.00	0.0000	20.000	100	71-129
Trichloroethene	20.00	0.0000	20.000	100	67-133
Trichlorofluoromethane	20.00	0.0000	20.000	100	48-152
Vinyl chloride	20.00	0.0000	20.000	100	4-196

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

NML = No Method Limits

COMMENTS: \_\_\_\_\_

## VOA Water Limits

	In-House	DOD Limits	South Carolina
1,1,1 Trichloroethane	80-125	65-130	70-130
1,1,1,2-Tetrachloroethane	70-140	80-130	70-130
1,1,2,2-Tetrachloroethane	70-130	65-130	70-130
1,1,2-Trichloroethane	80-130	75-125	70-130
1,1-Dichloroethane	75-130	70-135	70-130
1,1-Dichloroethene	70-125	70-130	70-130
1,2,4 Trichlorobenzene	45-135	65-135	70-130
1,2-Dibromo-3-chloropropane	70-130	50-130	70-130
1,2-Dibromoethane	75-130	80-120	70-130
1,2-Dichlorobenzene	70-130	70-120	70-130
1,2-Dichloroethane	70-135	70-130	70-130
1,2-Dichloropropane	75-130	75-125	70-130
1,3-Dichlorobenzene	65-125	75-125	70-130
1,4-Dichlorobenzene	70-125	75-125	70-130
2-Butanone	65-145	30-150	60-140*
2-Hexanone	70-140	55-130	60-140*
4-Methyl-2-pentanone	75-135	60-135	60-140*
Acetone	35-175	40-140	60-140*
Benzene	75-125	80-120	70-130
Bromochloromethane	80-125	65-130	70-130
Bromodichloromethane	85-135	75-130	70-130
Bromoform	70-140	70-130	70-130
Bromomethane	45-150	30-145	70-130
Carbon disulfide	65-130	35-160	70-130
Carbon tetrachloride	75-135	65-140	70-130
Chlorobenzene	75-120	80-120	70-130
Chloroethane	65-145	60-135	70-130
Chloroform	75-125	65-135	70-130
Chloromethane	45-145	40-125	70-130
Cis-1,2-Dichloroethene	80-120	70-125	70-130
Cis-1,3-Dichloropropene	75-130	70-130	70-130
Dibromochloromethane	80-140	60-135	70-130
Dibromomethane	65-140	75-125	70-130
Dichlorodifluoromethane	40-160	30-155	70-130
Ethylbenzene	75-130	75-125	70-130
Methylene chloride	70-130	55-140	70-130
M,p-Xylene	75-125	75-130	70-130
o-Xylene	75-130	80-120	70-130
Styrene	75-125	65-135	70-130
Tetrachloroethene	70-125	45-150	70-130
Toluene	75-125	75-120	70-130
Trans-1-2 Dichloroethene	70-125	60-140	70-130
Trans-1-3-Dichloropropene	70-130	55-140	70-130
Trichloroethene	80-125	70-125	70-130
Trichlorofluoromethane	70-140	60-145	70-130
Vinyl chloride	65-140	50-145	70-130
MTBE	75-125	65-125	70-130

Naphthalene	45-135	55-140		70-130
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\* - Poor Purger

### VOA Soil Limits

	Soil	DOD Limits		
1-1-1-2 Tetrachloroethane	75-125	75-125		
1-1-1-Trichloroethane	65-140	70-135		
1-1-2-2-Tetrachloroethane	65-130	55-130		
1-1-2-Trichloroethane	70-130	60-125		
1-1-Dichloroethane	70-135	75-125		
1-1-Dichloroethene	65-135	65-135		
1-2-4-Trichlorobenzene	55-135	65-130		
1-2-Dibromo-3-chloropropane	55-145	40-135		
1-2-Dibromoethane	70-135	70-125		
1-2-Dichlorobenzene	75-125	75-120		
1-2-Dichloroethane	65-140	70-135		
1-2-Dichloropropane	70-135	70-120		
1-3-Dichlorobenzene	70-125	70-125		
1-4-Dichlorobenzene	70-125	70-125		
2-Butanone	50-145	30-160		
2-Hexanone	50-140	45-145		
4-Methyl-2-pentanone	55-145	45-145		
Acetone	30-155	20-160		
Benzene	70-130	75-125		
Bromochloromethane	75-130	70-125		
Bromodichloromethane	70-145	70-130		
Bromoform	60-130	55-135		
Bromomethane	50-160	30-160		
Carbon disulfide	65-145	45-160		
Carbon tetrachloride	65-140	65-135		
Chlorobenzene	70-120	75-125		
Chloroethane	60-145	40-155		
Chloroform	65-135	70-125		
Chloromethane	50-150	50-130		
Dibromochloromethane	70-140	65-130		
Dibromomethane	75-125	75-130		
Dichlorodifluoromethane	35-155	35-135		
Ethylbenzene	75-125	75-125		
Methylene chloride	55-155	55-140		
Styrene	70-125	75-125		
Tetrachloroethene	65-130	65-140		
Toluene	55-140	70-125		
Trichloroethene	60-145	75-125		
Trichlorofluoromethane	60-140	25-185		
Vinyl Acetate	50-135			
Vinyl chloride	60-145	60-125		
cis-1-2-Dichloroethene	70-135	65-125		
cis-1-3-Dichloropropene	75-135	70-125		
m-p-Xylene	70-125	80-125		

o-Xylene	70-125	75-125		
trans-1-2-Dichloroethene	65-140	65-135		
trans-1-3--Dichloropropene	65-125	65-125		
MTBE	55-150			
Naphthalene	55-145	40-125		

**VOA MIXES**

LIQUID MIX (2000PPM)

C-610H (PRIMARY STANDARD)

M-502A-R-10X (ICV/LCS/MATRIX SPIKE)

Benzene  
 Bromobenzene  
 Bromochloromethane  
 Bromodichloromethane  
 Bromoform  
 n-Butylbenzene  
 sec-Butylbenzene  
 tert-butylbenzene  
 Carbon tetrachloride  
 Chlorobenzene  
 Chloroform  
 2- & 4-Chlorotoluene  
 Trichloroethene  
 Dibromochloromethane  
 1,2-Dibromo-3-chloropropane  
 1,2-Dibromoethane  
 1,2-Dichlorobenzene  
 1,3-Dichlorobenzene  
 1,4-Dichlorobenzene  
 1,1- & 1,2-Dichloroethane  
 1,1,2-Trichloroethene  
 1,1-Dichloroethene  
 cis-1,2-Dichloroethene  
 trans-1,2-Dichloroethene  
 1,2- & 1,3-Dichloropropane  
 1,2,3-Trichloropropane  
 2,2-Dichloropropane  
 1,1-Dichloropropene  
 cis-1,3-Dichloropropene  
 trans-1,3-Dichloropropene  
 Ethylbenzene  
 Hexachlorobutadiene  
 Isopropylbenzene  
 Isopropyltoluene  
 Methylene Chloride  
 Naphthalene  
 n-Propylbenzene  
 Styrene  
 1,1,1,2-Tetrachloroethane  
 1,1,2,2-Tetrachloroethane  
 Tetrachloroethene  
 Toluene  
 1,2,3-Trichlorobenzene

GASES MIX (2000PPM)

#30042 (PRIMARY STANDARD)

M-502B-R-10X (ICV/LCS/MATRIX SPIKE)

Bromomethane  
 Chloroethane  
 Vinyl chloride  
 Chloromethane  
 Dichlorodifluoromethane  
 Trichlorofluoromethane

C610H-PRIMARY STANDARD

M60310X (ICV/LCS/MATRIX SPIKE)

Acrolein (10,000PPM)  
 Acrylonitrile (10,000PPM)

CCS1037-PRIMARY CUSTOM MIX (5000PPM)

#557102- ICV/LCS CUSTOM MIX (5000PPM)

Tetrahydrofuran  
 MTBE  
 Methyl metacrylate  
 Ethyl methacrylate  
 1,1,2-Trichlorotrifluoroethane  
 Cyclohexane  
 Methylcyclohexane  
 Methyl acetate  
 Carbon disulfide  
 Iodomethane

PRIMARY and ICV/LCS STANDARD-neat

Vinyl acetate (5000PPM)

PRIMARY and ICV/LCS STANDARD-neat

2-Chloroethyl vinyl ether (20000PPM)

KETONES MIX (5000PPM)

#30006 (PRIMARY STANDARD and ICV/LCS) different lots

Acetone  
 2-butanone  
 2-hexanone  
 4-methyl-2-pentanone

1,2,3-Trimethylbenzene  
1,2,4-Trimethylbenzene  
1,3,5-Trimethylbenzene  
m-,p- & o-Xylene  
1,1,1-Trichloroethene

#### VOA CALIBRATIONS RANGE FOR EPA8260B/624(AQUEOUS)

Liquid mix, Gases mix & primary custom mix is calibrated at these levels - 0.5ppb, 1.0ppb, 2.0ppb, 10ppb, 20ppb, 50ppb, 100ppb & 200ppb. The ketones mix, 2-chloroethyl vinyl ether & vinyl acetate are calibrated at these levels - 1.0ppb, 2.0ppb, 4.0ppb, 20ppb, 40ppb, 100ppb, 200ppb & 400ppb. Acrolein & Acrylonitrile are calibrated at these levels – 2.5ppb, 5.0ppb, 10ppb, 50ppb, 100ppb, 250ppb, 500ppb, 1000ppb.

#### VOA CALIBRATIONS RANGE FOR EPA8260B(SOLIDS)

Liquid mix, Gases mix & primary custom mix is calibrated at these levels – 2.0ppb, 5.0ppb, 10ppb, 20ppb, 50ppb, 100ppb & 200ppb. The ketones mix, 2-chloroethyl vinyl ether & vinyl acetate are calibrated at these levels - 2.0ppb, 10ppb, 20ppb, 40ppb, 100ppb, 200ppb & 400ppb. Acrolein & Acrylonitrile are calibrated at these levels – 5.0ppb, 10ppb, 50ppb, 100ppb, 250ppb, 500ppb, 1000ppb.

#### VOA LCS/ICV/MATRIX SPIKE LEVEL FOR EPA8260B

Liquid mix, Gases mix, Ethyl methacrylate, Methyl methacrylate, Tetrahydrofuran, MTBE, Trichlorotrifluoroethane, Cyclohexane, Methylcyclohexane & Methyl acetate are spiked at 50ppb level. Acrolein & Acrylonitrile spiked at 250ppb level. Acetone, 2-Butanone, 2-Hexanone, Carbon disulfide, 2-Chloroethyl vinyl ether, 4-Methyl-2-pentanone, Vinyl acetate & Iodomethane are spiked at 100ppb level.

## INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE

COMPOUND	QUANT MASS	* I.S.	COMPOUND	QUANT MASS	* I.S.
*Fluorobenzene (1)	96		Dibromomethane	93	1
*Chlorobenzene-d5 (2)	117		1,1,2-Trichloroethane	83	2
*1,4-Dichlorobenzene-d4 (3)	152		1,2,3-Trichloropropane	110	2
Bromomethane	94	1	Hexachlorobutadiene	225	3
Chloroethane	64	1	Isopropylbenzene	105	2
Vinyl chloride	62	1	Isopropyltoluene	119	3
Chloromethane	50	1	Methylene Chloride	84	1
Dichlorodifluoromethane	85	1	Naphthalene	128	3
Acetonitrile	41	1	Propionitrile	54	1
Allyl chloride	41	1	n-Propylbenzene	91	3
Trichlorofluoromethane	101	1	Styrene	104	2
Benzene	78	1	1,1,1,2-Tetrachloroethane	131	2
Bromobenzene	156	3	1,1,2,2-Tetrachloroethane	83	3
Bromochloromethane	128	1	Tetrachloroethene	166	2
Bromodichloromethane	83	2	Toluene	92	2
Bromoform	173	2	1,2,3-Trichlorobenzene	180	3
n-Butylbenzene	91	3	1,2,4-Trichlorobenzene	180	3
sec-Butylbenzene	105	3	1,2,4-Trimethylbenzene	105	3
tert-butylbenzene	119	3	1,3,5-Trimethylbenzene	105	3
Carbon tetrachloride	117	1	m-Xylene	91	2
Chlorobenzene	112	2	p-Xylene	91	2
Chloroform	83	1	o-Xylene	91	2
Chloroprene	53	1	Acrolein	56	1
2-Chlorotoluene	91	3	Acrylonitrile	53	1
4-Chlorotoluene	91	3	Tetrahydrofuran	42	1
Dibromochloromethane	129	2	MTBE	73	1
1,2-Dibromo-3-chloropropane	157	3	Methacrylonitrile	41	1
1,2-Dibromoethane	107	2	Methyl methacrylate	41	1
1,2-Dichlorobenzene	146	3	Ethyl methacrylate	69	2
1,3-Dichlorobenzene	146	3	1,1,2-Trichlorotrifluoroethane	101	1
1,4-Dichlorobenzene	146	3	Cyclohexane	56	1
1,1-Dichloroethane	63	1	Methylcyclohexane	83	1
1,2-Dichloroethane	62	1	Methyl acetate	43	1
1,1-Dichloroethene	96	1	Carbon disulfide	76	1
cis-1,2-Dichloroethene	96	1	Iodomethane	142	1
trans-1,2-Dichloroethene	96	1	Vinyl acetate	43	1
trans-1,4-Dichloro-2-butene	53	3	2-Chloroethyl vinyl ether	63	1
1,2-Dichloropropane	63	1	Acetone	43	1
1,3-Dichloropropane	76	2	2-butanone	43	1
2,2-Dichloropropane	77	1	2-hexanone	43	2
1,1-Dichloropropene	75	1	Isobutyl alcohol	43	1
cis-1,3-Dichloropropene	75	1	1,4-Dioxane	88	1
trans-1,3-Dichloropropene	75	2	4-methyl-2-pentanone	43	1
Ethylbenzene	91	2	Dibromofluoromethane (S)	111	1
1,1,1-Trichloroethane	97	1	1,2-Dichloroethane-d4 (S)	102	1
Trichloroethene	95	1	Toluene-d8 (S)	98	2
			Bromofluorobenzene (S)	95	2

\*I.S.=internal Standard.

S=surrogate.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: 8260B/8270C (Circle One)</b>

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?	_____	_____	_____	_____
2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.( eg. m/p-xylene, ketones,etc.).	_____	_____	_____	_____
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?	_____	_____	_____	_____
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs.	_____	_____	_____	_____
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?	_____	_____	_____	_____
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?	_____	_____	_____	_____
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?	_____	_____	_____	_____
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?	_____	_____	_____	_____

Comments on any "No" response:

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Primary-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**GAS CHROMATOGRAPHY/ELECTRON  
CAPTURE DETECTOR (GC/ECD)**

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**ORGANOCHLORINE  
PESTICIDES/POLYCHLORINATED**

**BIPHENYLS (PCB) BY**

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**EPA METHOD 608/608.2 AND**

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**SW846 METHOD 8081A, 8081B/8082, 8082A**

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**SOP NUMBER:**

**SOP-211**

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**REVISION NUMBER:**

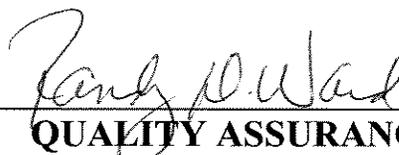
**20**

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**APPROVED BY:**

  
**SECTION MANAGER**

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**QUALITY ASSURANCE OFFICER**

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**EFFECTIVE DATE:**

**04/27/09**

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**Date of Last Review:**

**04/27/09**

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**GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD)  
ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB)  
BY EPA METHOD 608/608.2 AND SW-846 METHOD 8081A, 8081B/8082, 8082A**

## **1.0 SCOPE AND APPLICATION**

This Standard Operating Procedure, SOP, (based primarily on SW-846 Methods 8000B/8081A/8082) is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods SW-846 8082, *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. The normal laboratory list of analytes with their LCS limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Methods 8081A, 8081B/8082, 8082A. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8081A, 8081B/8082, 8082A, EPA 608/608.2 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, Quality Assurance Officer and/or Technical Director.

## **2.0 METHOD SUMMARY**

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition. Analytes are quantitated relative to known standards using the external standard method.

## **3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE**

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual includes details concerning sample preservation, containers and handling of samples and extracts. All water and soil samples are stored in the appropriate walk-in cooler in sample storage at a temperature of 1°C – 4.4°C. All extracts are stored in the Hobart in Semivolatiles laboratory at a temperature of 1°C – 4.4°C. Water samples have a holding time of 7 days from date of sampling to extraction. Soil samples have a holding time of 14 days from date of sampling to extraction (unless otherwise specified for the project). Extracts have a holding time of 40 days from extraction to analysis.

## **4.0 INTERFERENCES AND POTENTIAL PROBLEMS**

Section 3.0 of SW-846 Methods 8081A/8082 and Section 4.0 of Methods 8081B/8082A details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308, 309, 330, and 334) to evaluate possible clean-up options for any encountered interferences.

## **5.0 EQUIPMENT AND APPARATUS**

### **5.1 GC's:**

5.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.

## 5.2 Columns:

5.2.1 RTX-CLP (or equivalent): 30 meter x 0.32 mm ID x 0.5  $\mu$ m film thickness fused silica column.

5.2.2 RTX-CLP II (or equivalent): 30 meters x 0.32 mm ID x 0.5  $\mu$ m film thickness fused silica column.

## 5.3 Autosamplers:

5.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

5.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

5.5 Data Processing Software: Target DB Windows NT data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

## 6.0 REAGENTS

6.1 Hexane - pesticide quality or equivalent.

6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the GC standards logbook. The date they are opened is noted on the label and recorded in the GC standards logbook along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the GC standards logbook and given a sequential number. The following are noted in the logbook: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C-4.4°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook. See the GC standards log book for makeup of intermediate and calibration standards.

6.2.1 The Initial Calibration Verification (ICV) intermediate standard is prepared from the vendor stock standards in the same manner as the Calibration intermediate standards above and is stored in the refrigerator at a temperature of 1°C-4.4°C for up to 6 months. The ICV standard is then prepared at a concentration near the midpoint in the same manner as the Calibration standards above.

## 7.0 PROCEDURE

The GC/ECD should be primed by injecting a pesticide standard at 100  $\mu$ g/L and/or PCB standard at 10,000  $\mu$ g/L, 10 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

7.1 Chromatographic conditions:

- 7.1.1 ZB MR1/MR2 columns:
- |                                 |                       |
|---------------------------------|-----------------------|
| GC                              | ECD3                  |
| Purge on                        | 0.50 min.             |
| Injector/Detector temperature   | 250/350°C             |
| Column flow                     | @3.4 mL/min           |
| Initial column temperature      | 100°C for 0.5 minutes |
| Temperature ramp                | 15°C/min              |
| Intermediate column temperature | 220°C for 5.0 minutes |
| Second Temperature Ramp         | 35°C/min              |
| Final Column Temperature        | 340°C for 2.0 minutes |
- 7.1.2 RTX-CLP/RTX CLPII columns:
- |                               |                       |
|-------------------------------|-----------------------|
| GC                            | ECD4                  |
| Purge on                      | 0.50 min.             |
| Injector/Detector temperature | 200/330°C             |
| Column flow                   | @2.4 mL/min           |
| Initial column temperature    | 110°C for 0.5 minutes |
| Temperature ramp              | 15°C/min              |
| Final column temperature      | 320°C for 2.0 minutes |

7.2 Eval Mix – Before pesticide calibration and/or sample analysis, a degradation check standard (evaluation mix) of endrin and 4,4-DDT must be injected. Degradation of either compound must not exceed 15 percent. If 15 percent degradation is exceeded, then corrective action must be taken (GC system maintenance, see SOP-222).

7.3 Calibration - (See SW-846 Method 8000B Section 7.4.2).

7.3.1 Initial Calibration – For single component pesticides and surrogates, a six point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Technical Chlordane a single point standard is analyzed unless they are expected then a five point calibration is injected and analyzed. Injection volume for standards and samples is equal to 2 µL using the same injection technique to introduce both standards and samples (use of auto-injectors makes this a constant). All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. The percent relative standard deviation (RSD) of the calibration factor must be <20% over the working range for each analyte of interest. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient (r) is  $\geq 0.995$ ,  $\geq 0.99$  for quadratic with 6 points. Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other five Aroclor mixtures(1221, 1232, 1242, 1248 & 1254). The curves for Aroclors 1016 and 1260 are used to show the linearity of the detector and can be used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. The 1016/1260 curve may also be used to quantitate any 1016 or 1260 hits that may be seen in the samples. The analyst has the choice of running a curve for the other five Aroclor mixtures or when the calibration factor for the

curve for 1016/1260 is <20% a single point calibration at the midpoint level concentration can be used for the other Aroclors. The injection procedure for Aroclors is the same as single component pesticides. The percent relative standard deviation (RSD) of the calibration factor must be <20% but this applies to the average of the quantitation peaks. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient factor (r) is  $\geq 0.995$ ,  $\geq 0.99$  for quadratic with 6 points. Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

7.3.2 Initial Calibration Verification - A second source standard at the midpoint level is used to check the validity of the curve. The standard recovery for all analytes must be between 85 and 115% (**80-120% DOD QSM and 8081B/8082A**). If the second source recovery is above 115%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 85%, the second source standard may have deteriorated for that compound. This standard should be remade and reanalyzed. If this does not correct the problem, the standard should be replaced. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report.

7.3.3 Continuing Calibration Verification (CCV) - A mid-level standard must be analyzed every 12 hours (not to exceed 20 samples and cannot exceed 15 percent difference (%D), **(at the beginning and end of sequence and after every 10 field samples, 20%D no average DOD QSM and 8081B/8082A)**) from the average calibration factor of the calibration curve. A CCV must also be analyzed at the end of the analysis sequence. If a CCV fails, GC maintenance may be necessary (see SOP-222), reanalysis may be required (for samples analyzed since the last valid CCV) and a corrective action report must be completed. If none of the failed target compounds exceed 30% D and the average of all the %Ds are < 15% then the CCV may be used without any further corrective action. Alternatively, analytes may be flagged depending on their concentration and the status of the analyte in the mid-level standard. No reanalysis is necessary if the analyte is undetected in the samples and recovered high in the CCV. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. Samples are then quantitated against the initial calibration curve.

7.4 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration using the midpoint standard RTs. If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

7.5 Laboratory Control Sample (LCS) - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked with standards prepared from an alternate vendor or lot number than the calibration standards. See section 8.2 below for criteria and corrective action.

7.6 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch – up to 20 samples. See section 9.3 below for criteria and corrective action.

7.7 Samples - Prior to using Method 608, SW-846 8081A, 8081B/8082, 8082A or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3541, 3640, 3550, 3580, EPA method 608 or CLP).

7.7.1 Example of a sequence run log:

- 1-Primer A/B Mix-1000 or Primer PCB-10,000
- 2- EVAL Mix (Pest only)
- 3- CCV A/B Mix
- 4- CCV Toxaphene (single point)
- 5-CCV Chlordane (single point)
- 6- CCV PCB 1660
- 7- Method Blank
- 8-LCS A/B Mix
- 9-LCS PCB
- 10-Sample
- 11-Sample
- 12-Sample
- 13-Sample
- 14-Sample
- 15-Sample
- 16-Sample
- 17-Sample
- 18-Sample
- 19-Sample
- 20-Sample
- 21-Sample-MS
- 22-Sample-MSD
- 23-Sample
- 24-Sample
- 25-Sample
- 26-Sample
- 27-Sample
- 28-Sample
- 29- CCV A/B Mix
- 30-CCV PCB

7.8 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Target DB Windows NT data system. Quantitative measurements are performed as described in SW-846 8081A section 7.5.6, and section 11.5.6.1 8081B. Rounding is performed using CLP

odd/even rounding rules. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Formal data evaluation is detailed in SOP-216 (documented using the USACE Analyst Data Review Checklist for USACE projects).

7.8.1 Analyte concentration after rounding to 2 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

7.8.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

7.8.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your supervisor), [see SOP-308].

7.8.4 If the sample has extraneous peaks that are not removed by acid (PCB samples) and is not a sulfur interference, a florisol cleanup is recommended [see SOP-309]. A silica cleanup may also be used [see SOP-331].

7.8.5 Surrogates – Control and warning limits are determined by using all blank spikes in the calculation, (LCSs and method blanks). All limits used are generated in-house or client specified (with the exception that if the in-house limit's highest lower limit would be 90 and the lowest upper limit would be 110 examples: **98**-120 would be **90**-120 or 45-**75** would be 45-**110**). Surrogate standard recovery must be checked to determine if it is within these limits. Two surrogates are added to each sample for pesticides. Only one surrogate, DCB, is added to each PCB sample. Corrective action should be evaluated when any surrogate(s) is outside the action limits for a sample. When only one surrogate for pesticides exceed control limits on the primary and/or confirmation column corrective action may not be required. If both surrogates for pesticides (or DCB for PCB analyses) exceed control limits on the primary and/or confirmation column corrective action is required. A corrective action form should be filled out and given to the organic lab manager when both surrogates for pesticides (or DCB for PCB analyses) are outside the action or warning limits. The organic lab manager will then make suggestions as to what action should be taken, for example: the sample may need to be reanalyzed, reextracted, or flagged on the report for a QC problem. **DOD limits will be used for DOD QSM projects.**

	WATER	SOIL/SEDIMENT	WIPES
<u>Surrogate</u>	<u>In-House</u>	<u>In-House</u>	<u>In-House</u>
TCMX	25-120	30-120	80-143
DCB	25-130	35-140	73-142

7.9 Identification/Quantitation [See SW-846 method 8081A section 7.6 or method 8082 sections 7.7-7.9].

7.9.1 Single peak components are identified by retention time on a primary column

with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. The control limit for percent difference (%D) of compounds that confirmed from the primary column on the confirmation column is 40%. If both columns are equivalent, the highest concentration is reported. If a compound result is >40% difference, it should be flagged with a "P". ( **"J" flagged for DOD QSM projects**)

7.9.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be flagged appropriately.

7.9.1.2 If a compound is outside of it's window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to decide if the analyte is there.

7.9.2 Multi-peak components (PCB's, Toxaphene and Technical Chlordane) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 7.7.3 of SW-846 Method 8082. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.

7.9.2.1 Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components. Note: for USACE projects, five peaks are necessary for the quantitation of multi-peak components.

7.9.2.2 Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic lab manager's attention.

7.9.2.3 Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic lab manager's attention.

7.9.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response factor is 20% or less, then the concentration may be determined using the mean calibration factor, CF, from the initial calibration data. Otherwise, the analyst must use either a calibration curve or a

non-linear calibration model such as polynomial equation for quantitation. Calculations follow in Section 8.0. **Refer to SOP-224 for guidance for manual integrations**

## 8.0 Calculations:

8.1 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

8.2 The mean CF is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

8.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

8.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within  $\pm 15\%$  for each analyte before any sample analyses may take place.

8.5 Concentration in water samples is calculated as follows:

[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to  $\mu\text{g/L}$ .]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

where:

- $A_x$  = Area (or height) of the peak for the analyte in the sample.
- $V_t$  = Total volume of the concentrated extract ( $\mu\text{L}$ ).
- $D$  = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made,  $D = 1$ . The dilution factor is always dimensionless.
- $V_i$  = Volume of the extract injected ( $\mu\text{L}$ ). The nominal injection volume for samples and calibration standards must be the same.
- $CF$  = Mean response factor from the initial calibration.
- $V_s$  = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.

8.6 Concentration in non-aqueous samples is calculated as follows:

[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to  $\mu\text{g/kg}$ .]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

where:

- $A_x$ ,  $V_t$ ,  $D$ , and  $CF$  are the same as for aqueous samples, and
- $W_s$  = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.

## 9.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

9.1 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are charted to generate control limits and charts for diagnostic purposes. 8000B See section 7.8.5 above for criteria and corrective action.

9.2 LCS Sample - The LCS is extracted with every extraction batch - up to 20 samples. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number than the calibration standards. The recoveries are charted to generate control charts and limits. See the LCS report form in the appendix for the laboratory generated limits. These limits default to method limits if generated limits are wider. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only or results for that analyte should be flagged. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only. **DOD limits will be used for DOD QSM projects.**

9.3 Method Blanks - The concentration of all method target analytes should be below the MDL (**<RL, common laboratory contaminates; < 1/2 RL, all other compounds or client/authority specified**) for each method target analyte. If contamination exceeds the requirement, the following corrective actions must be taken. The first step is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 5x that found in the method blank.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for 608 projects, 1 in 10 samples are spiked for MS). For project specified full list MS/MSDs, the LCS standard is used for spiking. Criteria for the MS/MSD are found in Table 5-3 of the Laboratory Quality Assurance Manual. Both the percent recoveries (%R) and relative percent differences (RPDs) are contained in this table (for project specified full list MS/MSDs, the MS/MSD limits are the same as the LCS limits). Samples that do not meet these criteria due to matrix should be evaluated for placing a flag on the final report due to QC problems. The associated LCS results should be used to verify method performance (section 8.4.3 Method 8081A). MS data evaluation must include the consideration of the following factors.

9.4.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

9.4.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was more than four times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem. In this case, the MS/MSD may be evaluated as sample duplicates.

9.4.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

9.4.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

9.4.5 Documentation of capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## **10.0 HEALTH, SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION**

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

10.4 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **11.0 EXAMPLE FORMS**

Examples of the water and soil LCS report sheet and the USACE analyst data review checklist are located in the appendix.

**12.0 REFERENCES**

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846;* Method 8081A, 8081B, 8082, 8082A
2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136;* Method 608, 608.2; APX-B
3. *USEPA Contract Laboratory Program(CLP) for Organics ILM04.2; ILM04.3*
4. DOD Quality Systems Manual, Ver. 3/4.1

**13.0 DEFINITIONS**

Refer SOP-431 for a list of definitions.

## **APPENDIX**

FORM 3  
 WATER PESTICIDE/PCB LAB CONTROL SAMPLE  
 ( In- House)

Lab Name: Empirical Laboratories, LLC Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.: LCS

COMPOUND	SPIKE ADDED (µg/L)	SAMPLE CONCENTRATION (µg/L)	LCS CONCENTRATION (µg/L)	LCS % REC #	QC. LIMITS REC.
Aldrin	0.05000	NA	0.05000	100	25-110
alpha-BHC	0.1000	NA	0.1000	100	45-125
alpha-Chlordane	0.05000	NA	0.05000	100	50-125
beta-BHC	0.05000	NA	0.05000	100	50-130
4,4'-DDD	0.1000	NA	0.1000	100	55-130
4,4'-DDE	0.1000	NA	0.1000	100	45-130
4,4'-DDT	0.1000	NA	0.1000	100	50-140
Dieldrin	0.1000	NA	0.1000	100	55-130
delta-BHC	0.05000	NA	0.05000	100	40-135
Endosulfan I	0.1000	NA	0.1000	100	50-120
Endosulfan II	0.1000	NA	0.1000	100	55-135
Endosulfan sulfate	0.1000	NA	0.1000	100	55-130
Endrin	0.1000	NA	0.1000	100	40-150
Endrin aldehyde	0.1000	NA	0.1000	100	40-130
Endrin ketone	0.1000	NA	0.1000	100	60-130
gamma-BHC (Lindane)	0.1000	NA	0.1000	100	50-130
gamma-Chlordane	0.05000	NA	0.05000	100	50-125
Heptachlor	0.1000	NA	0.1000	100	35-125
Heptachlor epoxide	0.05000	NA	0.05000	100	50-130
Methoxychlor	0.5000	NA	0.5000	100	50-140
PCB-1248	10.00	NA	10.00	100	50-140

# Column to be used to flag recovery values with an asterisk

\* Values outside of QC limits

Spike Recovery: 0 out of 21 outside limits

COMMENTS: \_\_\_\_\_

EMPIRICAL LABORATORIES, LLC

SOP-211  
 Revision:18  
 Date: 01/24/08  
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FORM 3  
 SOIL PESTICIDE/PCB LAB CONTROL SAMPLE  
 (In-House)

Lab Name: Empirical Laboratories, LLC Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.: LCS

COMPOUND	SPIKE ADDED (µg/kg)	SAMPLE CONCENTRATION (µg/kg)	LCS CONCENTRATION (µg/kg)	LCS % REC #	QC. LIMITS REC.
Aldrin	5.0000	NA	5.0000	100	30-135
4,4'-DDT	10.000	NA	10.000	100	30-160
Dieldrin	10.000	NA	10.000	100	30-150
Endrin	10.000	NA	10.000	100	35-160
gamma-BHC (Lindane)	10.000	NA	10.000	100	30-145
Heptachlor	10.000	NA	10.000	100	40-150
PCB-1248	1000.0	NA	1000.0	100	50-150

# Column to be used to flag recovery values with an asterisk

\* Values outside of QC limits

Spike Recovery: 0 out of 7 outside limits

COMMENTS: \_\_\_\_\_

ANALYST DATA REVIEW CHECKLIST

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: 8081/8082</b>

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards?				
3. Is the low standard near, but above the MDL?				
4. Are the % RSDs within QC limits for all analytes?				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				

6. Are surrogate recoveries within QC limits? (one surrogate both \_\_\_\_\_ columns) \_\_\_\_\_

**ANALYST DATA REVIEW CHECKLIST**

- E. QC Samples
  - 1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs? \_\_\_\_\_
  - 2. Is the Laboratory Control Sample and its percent recovery within QC limits? \_\_\_\_\_
  - 3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits? \_\_\_\_\_
- F. Others
  - 1. Are all nonconformances included and noted? \_\_\_\_\_
  - 2. Are all calculations checked at the minimum frequency? \_\_\_\_\_
  - 3. Did analyst initial/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 they were performed correctly? \_\_\_\_\_

Comments on any "No" response:

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**TOTAL ORGANIC CARBON  
(TOC)**

**SM5310C,  
SW846 METHOD 9060/9060A  
AND LLOYD KAHN METHOD**

**SOP NUMBER:**

**SOP-221**

**REVISION NUMBER:**

**8**

**APPROVED BY:**

*Betty DeVill*  
**SECTION MANAGER**

*Randy D. Ward*  
**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:**

**04/28/09**

**DATE OF LAST REVIEW:**

**04/28/09**

**TOTAL ORGANIC CARBON (TOC)**  
**BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN**  
METHOD “*DETERMINATION OF TOC IN SEDIMENT*”

**I. SCOPE AND APPLICATION**

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060/9060A and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the Lloyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices. SW846 Method 9060/9060A and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

**II. SUMMARY OF METHOD**

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO<sub>2</sub> formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

**III. SAMPLING HANDLING AND PRESERVATION**

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory

indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

- 3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.
- 3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH  $\leq$  2) with HCl or H<sub>2</sub>SO<sub>4</sub>. Both water and soil samples are stored at 4°C.

#### **IV. INTERFERENCES**

##### **4.1 WATER METHOD**

- 4.1.1 Removal of carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.
- 4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

##### **4.2 SOIL METHOD**

- 4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.
- 4.2.2 The infrared detector is sensitized to CO<sub>2</sub> and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

## V. DEFINITIONS

- 5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.
- 5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.
- 5.3 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.
- 5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.
- 5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.
- 5.7 LABORATORY CONTROL SAMPLE (LCS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).
- 5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.
- 5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory

performance using test materials that have been prepared independently from the normal preparation process.

- 5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

## VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C ( $\pm$  2°C).

- 6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

- 6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

- 6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

- 6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

### 6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL  
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL  
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL  
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL  
25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL  
50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL  
100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL

0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL  
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL  
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL  
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL  
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL  
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

#### 6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.
2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

- 6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

- 6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

- 6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

## VII. INSTRUMENTATION

- 7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

- 7.2 There is a Shimadzu autosampler which will hold 68 samples.

- 7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

## VIII. AQUEOUS SAMPLE PROCEDURE

- 8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.
- 8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**
- 8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.
1. 100 ppm
  2. 50 ppm
  3. 25 ppm
  4. 10 ppm
  5. 5.0 ppm
  6. 2.5 ppm
  7. 1.0 ppm
  8. Method blank
  9. LCS + 9 samples (including any sample QC)
  10. 25 ppm
  11. 10 samples (including any sample QC)
  12. 50 ppm
- 8.8 Instrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

**TC Mode Instrument Settings:**

Analysis Temp: 900°C

Analysis Time: 6.5 minutes

Nitrogen Gas Flow: 60-100 psi (external regulator regulator)

**Oxygen Gas Flow: 40-60 psi (external regulator)**

**This is a step by step description of a routine soil TOC analysis.**

- 9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400 mL/minute and an oxygen flow of 180 mL/minute ( $\pm 3$  mL/minute).

**NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!**

- 9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.
- 9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).
- 9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.
- 9.5 If doing an initial calibration curve use an appropriate  $\mu$ L syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

$\mu$ L 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
50	15
100	30

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100  $\mu$ L of the 30% Sucrose standard with 500  $\mu$ L of water.

- 9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CC1+ date analyzed for ID) or Initial calibration – single analyses
  2. Method Blank(MB + date analyzed for ID) – single analyses
  3. LCS, 15 mg dextrose (LCS + date analyzed for ID) – single analyses
  4. NY Cert – 4 replicates
  5. Sample – 4 replicates
  6. Sample – 4 replicates
  7. Sample – 4 replicates
  8. Sample – 4 replicates
  9. Sample – 4 replicates
  10. CCV(CC1+ date analyzed for ID)2 – single analyses
  11. Sample – 4 replicates
  12. Sample – 4 replicates
  13. Sample – 4 replicates
  14. Sample – 4 replicates
  15. Sample – 4 replicates
  16. CCV (CC2+ date analyzed for ID) – single analyses
  17. Sample – 4 replicates
  18. Sample – 4 replicates
  19. Sample – 4 replicates
  20. Sample – 4 replicates
  21. Sample – 4 replicates
  22. CCV(CC3+ date analyzed for ID) – single analyses
  23. Sample – 4 replicates
  24. Sample – 4 replicates
  25. Sample – 4 replicates
  26. Sample – 4 replicates
  27. Sample – 4 replicates
  28. CCV(CC4+ date analyzed for ID) – single analyses
  29. SampleMS – 4 replicates
  30. SampleDUP – 4 replicates
  31. FCV(CC4+ date analyzed for ID) – single analyses
  32. FCB(FCB4+ date analyzed for ID) – single analyses
- 9.7 Samples should be stored away from light and at 4°C ( $\pm$  2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.
- 9.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are

exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot is taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.**

- 9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.
- 9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## X. QC REQUIREMENTS

- 10.1 Analyze a laboratory control sample (LCS) for each batch of samples **(maximum of 10 samples per day)**. If the LCS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.
- 10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.
- 10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.
- 10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are  $\leq 15\%$  for aqueous samples and  $\leq 20\%$  for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or  $\leq 30\%$  (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

- 10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.
- 10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than  $\pm$  the MDL.
- 10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.
- 10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

- 10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within  $\pm 10\%$ . A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060/9060A requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required number of sample replications.
- 10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

## **XI. CORRECTIVE ACTIONS**

### **11.1 INSTRUMENT RELATED**

1. ICV not within  $\pm 20\%$  (Soil) or  $\pm 10\%$  (SM 5310C0)
  - a. If the problem is with the solution.

- i. Re-prepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within  $\pm 30\%$  (Soil) or  $\pm 15\%$  (SM 5310C)
  - a. If the problem is with the solution.
    - i. Re-prepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalyzed.

**\* Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

#### **11.2 SAMPLE MATRIX RELATED**

1. Replicate analysis RPD not within  $\pm 20\%$  aqueous or  $\pm 50\%$  soil/sediment
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$  aqueous or  $\pm 50\%$  soil/sediment
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

## **XII. HEALTH AND SAFETY**

- A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.
- D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

## **XIII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

**XIV. METHOD PERFORMANCE**

**14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)**

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

\*KHP = potassium acid phthalate.

14.2 There was no method performance data available for the soil procedure.

**XV. REFERENCES**

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).
3. EPA SW-846, Method 9060/9060A.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

**APPENDIX I.**

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eighth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1" beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they are loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightning bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into correct position will take almost

- 3 hours to finish. Can view data as its coming off by selecting “view” “sample window”. After calibration is done review.
27. Select “File” then “New” then “sample run” “ok”
  28. General information screen: No change select “ok”
  29. Save as screen: Select today’s date for file name example 00month/00day/00year
  30. Select “save”
  31. Sample Table Screen: Select “insert” then select “ auto generate” enter
  32. **Page 1** sample group wizard sample source: select “calibration curve” then double click on box with 3 dots ...
  33. Open latest curve from calibration curves file
  34. Highlight latest curve and select “open”
  35. Should send you back to page 1 with calibration curve info submitted. Select “next”
  36. **Page 2** Sample Parameter: Enter final sample count for “number of samples” select “next”
  37. **Page 3** Calibration Curves: No changes Select “Next”
  38. **Page 4** Calibration Checks: No changes Select “Next”
  39. **Page 5** Controls: No changes select “finish”, Select “ok” on “Sparging/ Acid page.
  40. Type sequence as they are loaded on tray: ICV, ICB, LCSW, Sample #, client,etc.
  41. Once everything is typed in double check that it matches the way samples and QC are loaded..
  42. Click or select the lightening bolt symbol then select “use settings on PC”. Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
  43. Click or select “shut down Instructions”. Then select “standby” Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select “OK” if it looks the same.
  44. Start ASI measurement: External acid addition should have a check mark click on “start” analysis should begin to start.
  45. Click on view and chose “sample window” to watch curves come off and to see beginning values.

**GC/MS VOLATILE  
NON-AQUEOUS MATRIX  
EXTRACTION USING  
SW-846 METHOD 5035  
FOR 8260B ANALYSIS**

**SOP NUMBER:**

**SOP-225**

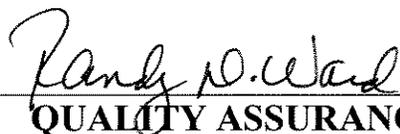
**REVISION NUMBER:**

**8**

**APPROVED BY:**



**SECTION MANAGER**



**QUALITY ASSURANCE  
MANAGER**

**EFFECTIVE DATE:**

**09/24/08**

**DATE OF LAST REVIEW:**

**09/24/08**

**GC/MS - VOLATILE  
NON - AQUEOUS MATRIX EXTRACTION  
USING SW-846 METHOD 5035**

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035. Soil samples should be sampled in the field using the EnCore™ sampler then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

## **2.0 SUMMARY**

Samples are collected in EnCores or Terracore vials. EnCore samples have to be prepped within 48 hrs of collection. Terracores are shipped already prepared.

## **3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE**

EnCores are prepped within 48 hrs of collection in sodium bisulfate and refrigerated at 4°C or in reagent water and frozen. Terracores are prepped in the field in sodium bisulfate or water. Sodium bisulfate Terracores are refrigerated at 4°C and those prepped in reagent water are frozen. Holding Time is 14 days

## **4.0 INTERFERENCES AND POTENTIAL PROBLEMS**

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

## **5.0 EQUIPMENT AND MATERIALS**

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

## 6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH<sub>3</sub>OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, NaHSO<sub>4</sub> – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of NaHSO<sub>4</sub> (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the NaHSO<sub>4</sub> in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

## 7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

**7.1** The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be

determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

- 7.2** All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the “low-level” sample. (Some projects may not require the “low-level” detection limits, in this case only the high level sample preparation would be required.) A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. Additional bulk samples should be collected for screening and dry weight determination without the preservative. Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

## **8.0 PROCEDURE**

- 8.1** Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.
- 8.2** The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.
- 8.3** Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:
- 8.3.1** Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.

- 8.3.2** Get the samples from the Hobart assigned for volatile sample storage and log them out.
- 8.3.3** Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).
- 8.3.4** Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within  $\pm 0.01g$  of this value before using for sample preparation.
- 8.3.5** For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate a CAR to document the problem.

- 8.3.6** Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.

- 8.3.7** Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

## **9.0 ANALYSIS**

- 9.1** Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.
- 9.2** Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

## **10. HEALTH, SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION**

- 10.1** Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- 10.2** Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
- 10.3** MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.
- 10.4** Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## REFERENCES

*1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035.*

## DEFINITIONS

Refer to SOP-431 for common environmental laboratory definitions.

**Methane, Ethane, Ethene in Aqueous  
Samples by Modified RSK-175  
(Automated Headspace)**

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**SOP NUMBER:**

**SOP-236**

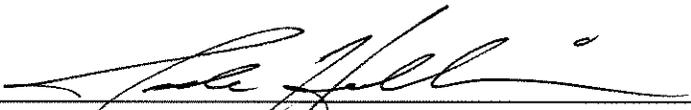
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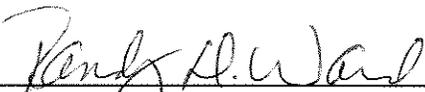
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**APPROVED BY:**

  
**SECTION MANAGER**

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**QUALITY ASSURANCE MANAGER**

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**EFFECTIVE DATE:**

**05/23/08**

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**DATE OF LAST REVIEW:**

**04/28/09**

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## **Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175 (Automated Headspace)**

### **I. SUMMARY**

The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples. Reporting limits for these are methane 2.0 ug/L, ethane 1.4 ug/L, and ethene 1.1 ug/L.

### **II. SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE**

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. Samples are collected in 40 ml VOA vials and shipped to the lab in coolers with ice. Water samples are stored in the Hobart in the sample storage room at a temperature of 4°C.

### **III. INTERFERENCES AND POTENTIAL PROBLEMS**

Methane found in the lab environment can be a source of contamination. The blank value is subtracted from the sample results.

### **IV. INSTRUMENTATION AND EQUIPMENT**

- A. Gas Chromatograph
  - 1. HP 5890 Series II (temperature programmable).
- B. Autosampler
  - 1. Tekmar 7000 Headspace autosampler
- C. Columns-Capillary columns.
  - 1. Carboxen 1006 PLOT column—30 meter x 0.53mm ID
- D. Data Acquisition and Processing Software.
  - 1. HP Chemstation system is interfaced to the HP-GC for data acquisition and storage.
  - 2. TARGET data system is interfaced to the acquisition systems. The system accepts, processes and stores acquired data.
- E. Glassware
  - 1. 25ml Graduated cylinder
  - 2. 20ml headspace vials with crimp tops( National Scientific)

3. Gastight syringes- 25, 50, 100, and 250uL

## V. STANDARDS

Gas standards are purchased from Restek and Supelco. The date they are received is noted on the container they are received in. The standards are given a sequential number the day they are opened and this is noted in the GC standards logbook. Standards for MEE are Scotty gases purchased in pressurized tanks. Calibration standards at a minimum of five levels are prepared by injecting the gas from a Scotty gas standard tank into capped 20 ml headspace vials with 15 ml of D.I. water using a gas-tight syringe. Usually 5,10,20,25,50,100,150,200ul and up to 5ml are used.

## VI. PROCEDURE

The following information describes the instrument and QC requirements to analyze the compounds that we do by this method.

### A. Instrumentation

#### 1. GC

- Initial Temperature: 35 ° C hold for 3.0 minutes.
- Ramp: 25 °C / minute to 225 ° C.
- Final Temperature 225 ° C hold for 3.08 minutes.
- Detector Temperature 230 ° C.

#### 2. Headspace Autosampler

- Platen: 80 °C./ Platen Equil.: 0.50 min.
- Sample Equil.: 2.0 min.
- Pressurization: 0.50 min./ Pressure Equil.: 0.25 min.
- Loop Equil.: 0.30 min.
- Injection Time: 1.0 min.
- Valve and Line Temp.: 95°C.

### B. Calibration and Quality Control

1. Refer to SW-846 Method 8000B for proper calibration techniques.
  - a. Five point minimum calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 percent over the working

range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve (linear curve corr.  $\geq 0.995$ , quadratic  $\geq 0.99$  with six points). The curve is then verified using a second source standard (**75-125% criteria**).

- b. The calibration curve must be verified every day through the analysis of a mid-level standard at the beginning and end of the sequence and after every 10 field samples. The percent difference back to the curve must not exceed  $\pm 20$  percent. If this criteria is not met, corrective action must be taken before sample analyses continues. Usually this involves recalibration or checking the gastight syringes.

c. Calculations:

$$\text{Calibration Factor (CF)} = \frac{\text{Response}}{\text{Dec Equiv} \times 1000}$$

Decimal equivalents are taken from the sample quant reports and entered into an Excel spreadsheet to calculate final concentration in ug/L.

2. Retention Time (RT) Windows - RT criteria set forth in SW-846 method 8000C are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration with midpoint standard. If the established retention time window is less than  $\pm 0.03$  minutes, the window defaults to  $\pm 0.03$  minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.
1. Quality control for this method can be referenced in SW-846 Method 8000C.
  - a. A method blank is required before analyzing samples. The contamination level should not exceed the CRDL.
  - b. An MS/MSD pair are required every 20 samples per matrix. Limits 75-125%.
  - c. A Laboratory Control Sample (LCS) is required every 20 samples. Limits 75-125%.
  - d. MDLs are either performed annually or by analyzing an MDL check according to SOP-414.

- C. Sample analysis includes the following steps: 15 ml of sample are transferred to 20ml headspace vials capped and loaded onto the autosampler along with a method blank with 15 ml of D.I. water.
1. A mid-level standard must be run at the beginning and end of the sequence and after every 10 field sample and cannot exceed  $\pm 20$  percent difference from the initial calibration. A mid-level standard must also be analyzed at the end of the analysis sequence.
  2. The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples.
- D. Following sample analysis, the data is reduced using the TARGET data system. The following must be checked to see if the samples will require re-analyses or dilution.
1. The analyte concentration must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.
- E. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with these methods. See SOP-413 for guidance.

## VII. HEALTH AND SAFETY

- A. Care should be used in handling all samples.
1. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
  2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
  3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

## VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION

A. Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## IX. REFERENCES

- A. Newell, Bryan, RSKSOP-175, Rev.0, August 1994.
- B. Newell, Bryan, RSKSOP-147, Rev.0, January 1993.
- C. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8000C.

**DEFINITIONS**

°C - degrees centigrade  
CF - calibration factor  
CRDL - contract required detection limit  
%D - percent difference  
FID - flame ionization detector  
GC - gas chromatograph  
LCS - laboratory control sample  
MDL - method detection limit  
µL - microliter  
µm - micrometer  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
%RSD - percent relative standard deviation  
RT - retention time  
SOP - standard operating procedure

Refer to SOP-431 for further definitions.

GC/MS SEMI-VOLATILE  
BNA-AQUEOUS MATRIX  
EXTRACTION USING  
SW-846 METHOD 3510C  
FOR 8270C/625 ANALYSIS

**SOP NUMBER:**

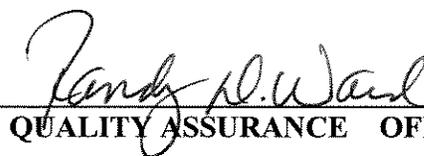
SOP-300

**REVISION NUMBER:**

17

**APPROVED BY:**

  
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QUALITY ASSURANCE OFFICER

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09/23/08

**GC/MS BNA - AQUEOUS MATRIX EXTRACTION  
USING SW846 METHOD 3510C/8270C, 625****I. SCOPE AND APPLICATION/SUMMARY**

1. This SOP describes the extraction of BNAs from water by separatory funnel extraction using SW846 Method 3510C and 625. Samples are extracted with methylene chloride and concentrated to an appropriate final volume.

**II. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**III. APPARATUS AND MATERIALS**

- Separatory Funnel - 2-Liter with Teflon stopcock
- Beaker - 400 mL
- Drying /Chromatographic column - 20 mm I.D. x 300 mm or funnel
- Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- Metal rack - capable of holding six glass evaporation tubes
- Turbo-Vap Evaporator - heated and capable of temperature control ( $\pm 5^{\circ}\text{C}$ ); the bath should be vented into a hood.
- Vials - 2 mL glass amber, with Teflon-lined screw cap and 40 mL with Teflon lid.
- pH indicator paper - close range (1.0 - 2.0) and (10.0 - 12.0); wide range (1.0 - 12.0)
- Syringe - 1 mL, 500 mL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Ring stand - 3 prong
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator

**IV. REAGENTS**

- Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.
- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
- Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in plastic bulk containers from Fisher # S415-10S or equivalent) placed in Pyrex tray and heated at 400°C for a minimum of 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.
- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).
- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H<sub>2</sub>SO<sub>4</sub> (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.
- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent).
- The GC/MS operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

**BNA Surrogate** - The base neutral and acid surrogates are normally mixed together in one solution. This solution is purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for surrogate amount of 100:200 ug/mL per sample. (**For low level PAHs use 1.0ml of a 1.0µg/mL BN Surrogate spiking solution.**)

**BNA Spiking Solution** - The base neutral and acid spiking solutions are normally mixed together in one solution ( **This spiking solution contains all the compounds that are normally calibrated by GC/MS** ). This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for LCS amount of 100ppm per sample. There are two separate spiking solutions available – one solution has a more complete list of BNA compounds than the other which is called the short or matrix spike list. The long list should be used on all extractions unless your supervisor has approved the short list. The short list may be used for any ‘phenol only’ extractions. (**For low level PAHs use 1.0 ml of a 1.0ppm of the LLPAH spiking solution.**)

**BNA TCLP Spike** – 0.5 mL is added per 100-mL volume. Each matrix type must have its own TCLP spike. TCLP spike should be added after the TCLP has been filtered but prior to refrigeration. From the volume provided by Wet Chemistry, remove a 100-mL aliquot into a suitable container with a teflon lid, and spike as indicated above.

## V. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
  - Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
  - Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
  - Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
2. Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

**SLUDGE** - use only 100 mL and dilute to 1000 mL with reagent water.

**TCLP EXTRACT** - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (which has already been spiked as explained in the BNA TCLP Spike section above) should be set up at the same time. Dilute to 1000 mL with reagent water.

**BAD MATRIX** – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. **SPLP extract- use 1 liter.**

**NPDES client** - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1 mL is added to the LCS and the matrix spike.

**ACID EXTRACT WITH BAD MATRIX** - a cleanup step is added. Samples are taken to a high pH, extracted with 60 mLs methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

3. **LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs)** – Samples require a BNA extraction. Use the Surrogate and BN spiking solutions specified. Low level PAHs are normally concentrated on the Turbo-Vap using Round-Bottom TV tubes to a final volume of 0.5 mL
4. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.
5. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume-Final Volume, and the Lab prebatch code. The lab batch code is defined as MMDDYYB# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.
6. Using the 1000-mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000 mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.
7. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

Generally 0.5 mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. **For low level PAHs use 1.0ml of a 1.0ppm LLPAH Surrogate spiking solution.** Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

For the sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe designated for BNA spike, to add 0.5 mL of BNA spiking solution. **For low level PAHs use 1.0 mL of the 1.0ppm LLPAHs spiking solution.** Someone must verify that the spike has been added by placing a check mark on each label as it is added. **For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.**

Enter the ID# of the surrogate/spike used and the initials of the person that verified their addition to the sample in the BNA logbook.

8. **ACID EXTRACTION:** Adjust the pH to between 1.0 and 2.0, using 2 mL of 1:1 H<sub>2</sub>SO<sub>4</sub>. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H<sub>2</sub>SO<sub>4</sub> solution in small increments, as required to attain the proper pH.
9. Add 40 mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.
10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.

**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.**

11. Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.
12. Following Steps 9 and 10, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.
13. **BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater**, using 10N NaOH. Start by adding 5.0 mLs to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. **BNA extraction is necessary when doing low level PAHs.**

**NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.**

14. **FOR 8270 extraction:** Extract one more time with 40 mL of methylene chloride following Steps 9 and 10. Combine BN and Acid extracts in a same 400ml beaker, unless

the BN extract has large amount of emulsion; then it will be necessary to use a separate 400 mL beaker. Concentrate BN and acid extracts for one final extract.

**NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.**

**For 625 extraction:** extract 3 more times with 40 mL methylene chloride following steps 9 and 10. Combine BN extracts in separate 400 mL beaker. Concentrate BN and acid extracts separately for one final extract.

15. In the log book marked BNA extractions, enter the Client name, the Lab #, the date extracted, the initial volume, and 1.0 mL for the final volume and anything unusual that may have occurred with this sample. The final volume for low level PAHs is 0.5 mL.
16. Prepare to dry the sample by either of the following methods:
  - 16A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR
  - 16B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.
17. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.
18. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of

Na<sub>2</sub>SO<sub>4</sub> by sprinkling the top layer with Na<sub>2</sub>SO<sub>4</sub> until it hardens, separates, and drops to the bottom.

## 19. TURBO-VAP CONCENTRATION

Low level PAH sample concentration is primarily done by Turbo-Vap using Round-Bottom TV tubes.

- Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.
- Record the numbers of the Turbo-Vap tube in the BNA logbook and remove the tube to a metal holder. To help prevent cross contamination, place a piece of aluminum foil over the Turbo-Vap tube and punch a small hole in the top so that the nitrogen can be accessed.
- Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.
- Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. **THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!!** A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.25 mL of methylene chloride into a 0.50 mL syringe and add this aliquot to the centrifuge tube. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2-mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Compare the volume to a 2mL dummy vial containing 0.5 mL of

solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

20. Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume in the BNA logbook and the Avery label to the nearest 10-mL.
21. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

#### **VI. DOCUMENTATION OF CAPABILITY ( DOC)**

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

#### **VII. WASTE MANAGEMENT AND POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### **VIII. METHOD PERFORMANCE**

Refer to SOP-201 for method performance.

#### **IX. HEALTH AND SAFETY**

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

#### **REFERENCES**

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 625.

#### **DEFINITIONS**

BNA- base/neutral acid  
°C - degrees centigrade

**EMPIRICAL LABORATORIES, LLC**

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COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample  
 $\mu\text{g/L}$  - micrograms per liter  
 $\mu\text{L}$  - microliter  
 $\mu\text{g/ml}$  - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
PAH- polynuclear aromatic hydrocarbon  
RL - reporting limit  
SOP - standard operating procedure  
v/v - volume to volume

Refer to SOP-431 for further definitions

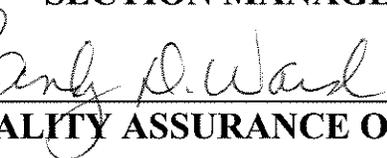


**PESTICIDE/PCBs**  
**AQUEOUS MATRIX EXTRACTION**  
**FOR EPA METHOD 608/608.2 AND**  
**SW846 METHOD 8081A/8082**  
**USING SW846 METHOD 3510C**

**SOP NUMBER:** SOP-302

**REVISION NUMBER:** 16

**APPROVED BY:**   
**SECTION MANAGER**

  
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**PESTICIDE/PCB - AQUEOUS MATRIX EXTRACTION  
BY EPA METHOD 608/608.2 AND SW-846 METHOD 8081A/8082  
USING SW846 METHOD 3510C**

**I. SCOPE AND APPLICATION**

1. This SOP describes the extraction of pesticides/PCBs from water by separatory funnel extraction using SW846 Method 3510C and Method 608/608.2.

**II. SUMMARY**

1. Aqueous samples are extracted three times with methylene chloride. The extracts are dried through sodium sulfate and concentrated and exchanged to hexane.

**III. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**IV. APPARATUS AND MATERIALS**

- Separatory Funnel - 2 Liter with Teflon stopcock
- Beaker - 400 mL
- Drying/Chromatographic column - 20 mm I.D. x 300 mm (or funnel)
- Turbo-Vap evaporation tube - 200-mL tube made by Zymark to fit into Turbo-Vap evaporator.
- Metal rack - capable of holding six glass evaporation tubes.
- Turbo-Vap Evaporator - heated and capable of temperature control (+5°C); the bath should be vented into a hood.
- Vials 10-mL glass, with Teflon-lined screw cap
- pH indicator paper - wide range (1.0 -12.0)
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Volumetric Flasks - Class A, glass, 1000 mL, 500 mL, 100 mL, 50 mL.
- Ring stand - 3 prong

- Burette clamp - double
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator
- Boiling chips - Teflon

## V. REAGENTS

- Reagent water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.
- Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>) - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in bulk containers from Fisher # S415-10S or equivalent] placed in a Pyrex tray and heated at 400°C for 4 hrs minimum, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.
- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).
- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H<sub>2</sub>SO<sub>4</sub> (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.
- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent)
- Hexane - suitable for gas chromatography (Omnisolv HX0298-1 or equivalent)
- The GC operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

TCMX/DCB (2, 4, 5, 6 - Tetrachloro-meta-xylene/Decachlorobiphenyl) - Surrogate solution is prepared in acetone by making a cut on stock purchased from a reputable vendor. The final concentration is 0.5 µg/mL. Generally use 1.0 mL of this solution per 1000ml of aqueous sample.

PCB Spiking Solution - The PCB of choice (1242, 1248, 1254, or 1016/1260 are the most common) is prepared in acetone at a concentration of 5.0 PPM. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Generally use 0.2- 1.0 mL per 1000ml of aqueous sample.

Pesticide Spiking Solution - A spiking solution is prepared at the appropriate concentration of 1 ppm. Generally use 1 mL per 1000ml of aqueous sample. For 608 samples, 1 out of every 10 samples must be spiked.

- TCLP's - When necessary to set up a TCLP, in addition to setting up the sample, two matrix spikes must be set up and should include the following: 1.0 mL is added per 100 mL volume. Each matrix type must have its own TCLP spike.

TCLP Spike 1 - includes Chlordane at 10 µg/mL and Toxaphene at 10 µg/mL. These spikes are made up individually and are also used as the LCS spike. Add 1 mL of each to a 100-mL aliquot.

TCLP Spike 2 - includes Mix A and Mix B @ 100 to 1000 µg/L ppb (also used as the pesticide spiking solution). These spikes are combined into one solution. Add 1 mL per 100-mL aliquot.

## VI. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract as follows:

Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.

Samples requiring RUSH turn around time may be logged in throughout the day that will require your immediate attention. Login personnel will generally communicate this need.

Check with login throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

2. Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

SLUDGE - use only 100 mL and dilute to 1000 mL with reagent water.

TCLP EXTRACT - use only 100 mL for the sample and dilute to 1000 mL with reagent water. Two matrix spikes of 100 mLs (per client matrix). Dilute to 1000 mL with reagent water.

BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any, should be made.

NPDES client - Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0 and 9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9 using NaOH solution or H<sub>2</sub>SO<sub>4</sub>, as necessary) or to extract the sample within 72 hours of sampling. If a pH adjustment is made, the details of the adjustment must be recorded on the sample chain of

custody and the outside of the sample bottle. Set up one full list matrix spike for every ten samples.

3. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook
4. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS are to be processed with each set of samples. If the sample is a TCLP, a blank may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples) except for TCLPs and 608 samples as noted above. In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code. The lab prep batch code is defined as MMDDYY# where #: 1 = 1st Method Blank for the day; 2 = 2nd Method Blank for the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.
5. Using the 500 mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1 liter of reagent water from the carboy and transfer it to a separatory funnel for the Method Blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Add 50 mL of Methylene Chloride to the empty bottle.
6. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

Using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 1.0 mL of TCMX/DCB surrogate to each sample, blank and spike. A second analyst must verify that the surrogate and spike has been added. Enter the ID# of the standard, amount, and the initials of the analysts in the extraction logbook.

7. Determine if the sample will require a Pesticide Spike or a PCB Spike or both. Proceed as follows:

Pesticide and PCB - set up two LCS's – one for Pesticide getting a MIX A&B spike and one for PCB, which should be spiked with PCB 1016/1260. In addition to the LCS's, a matrix sample spike and duplicate is spiked for pesticides and PCBs.

Pesticide only – To the sample in each analytical batch selected for spiking, add 1.0mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

PCB only - To the sample in each analytical batch selected for spiking, add 1.0 mL of PCB 1016/1260 or 1248 (project specific) unless otherwise specified using a 1.0-mL glass syringe dedicated to that PCB.

NOTE: Due to limited volume received, usually it is necessary to use 1/2 liter to do a spike so that a spike duplicate can be extracted also. If only one liter is provided for spiking purposes, use a 500-mL glass cylinder to measure out half the sample. Transfer to a separatory funnel labeled for the Spike. Measure the remaining sample and transfer to a separatory funnel labeled Spike Duplicate. Add 1/2 the normal amount of spiking solution and 1/2 the normal amount of surrogate.

Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the extraction logbook.

8. If necessary, adjust the pH to between 5.0 and 9.0 using NaOH solution or H<sub>2</sub>SO<sub>4</sub>. If a pH adjustment is made, the details of the adjustment must be recorded in the extraction logbook.
9. Swirl the 50-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel. In addition, add 50 mLs of methylene chloride to the method blank, LCS, and spikes.
10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, teflon separatory funnels may be placed in the shaker apparatus with the stopcock slightly open. When this apparatus is used, the shake should be for 3 minutes.

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.

11. Allow the sample to set for 10 minutes, if needed, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 50 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.
12. Following Steps 10 and 11, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.
13. In the logbook marked Pest/PCB extractions, enter the Client name, the Lab #, the date extracted, and anything unusual that may have occurred with this sample.

14. Dry the sample by either of the following methods:
  - 14.1 Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with of methylene chloride 20-30 mL and discard this rinse.
  - 14.2 Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it. Add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse.
15. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the orange holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary, use a dummy bottle with similar weight using reagent water. Set the rpm at 2500 and the temperature at 25°C. Close the lid and be sure to press it down until you hear it lock. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.
16. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer. Use the smallest amount possible of Na<sub>2</sub>SO<sub>4</sub> by sprinkling the top layer with Na<sub>2</sub>SO<sub>4</sub> until it hardens, separates, and drops to the bottom.
17. TURBO-VAP CONCENTRATION
  - 17.1. Rinse a Turbo-Vap tube and arrange it underneath a rinsed, packed drying column/funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker twice with 10 mL of methylene chloride. Rinse the column with 10 to 15 mL of methylene chloride. For solvent exchange purposes add 50 mL of hexane to each tube. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. While waiting for the drying column/funnel to quit dripping, record the numbers of the Turbo-Vap tube in the Pesticide/PCB logbook and remove the tube to a metal holder.

- 17.2 Turbo-Vap Operation: Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. Temperature of the bath should be at 40°C -50°C. To help prevent cross contamination, cover the turbovap tube with aluminum foil and punch a small hole in the top.
- 17.3 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- 17.4 When the Pesticide and/or PCB samples concentrate to approximately one mL, remove the tube from the Zymark.

NOTE: **For PCBs Only\*** Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows: Add just enough methylene chloride to make the gel go back into solution. Acid clean the extract and reconcentrate. Exchange with hexane again. If gel forms again, add enough methylene chloride to get gel back into solution. Transfer to a suitable container and record the final volume on the label and the extraction logbook. Note the percentage of methylene chloride in sample also.

- 17.5 When the beep sounds indicating the end of concentration, the extract will be at approximately one – one half mL (half way up tip of tube). Remove the tube from the bath. Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 4.0-mL vial. Spilling a drop or two during the transfer is critical because one drop represents 5% of the sample.
- 17.6 Add 1 ml of hexane to the turbovap tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 4.0-mL vial. Rinse again with 1 ml of hexane and transfer to the sample vial. Adjust the volume to 4.0 ml with hexane. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.
18. The extract obtained above may now be analyzed. Refrigerate to 4°C. Samples must be signed into the Hobart refrigerator. On log provided, enter the sample numbers, analyst initials, and the date and time the samples were placed into the Hobart.

**NOTES:**

If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Refer to the Florisil Cartridge Cleanup Method, the Copper Cleanup Method, and/or the Acid Cleanup Method for further information. (SOP-309, 307, and/or 308)

Pesticides that have aldrin detected in them may be cleaned using the SW-846 method 3630 (Silica Gel Cleanup). The GC Lab would notify extraction personnel if this cleanup is necessary.

#### **VII. DOCUMENTATION OF CAPABILITY (DOC)**

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

#### **VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### **IX. METHOD PERFORMANCE**

Refer to SOP-211 for method performance.

#### **X. HEALTH AND SAFETY**

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

#### **REFERENCES**

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 608.

#### **DEFINITIONS**

°C - degrees centigrade  
COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample  
µg/L - micrograms per liter  
µL - microliter  
µg/ml - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
PCBs - polychlorinated biphenyls  
Pest - pesticides  
RL - reporting limit  
SOP - standard operating procedure  
TCMX - tetrachloro-m-xylene  
v/v - volume to volume

Refer to SOP-431 for further definitions

**HERBICIDES AQUEOUS**

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**MATRIX by METHOD EPA SW-  
846 8151A**

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**SOP NUMBER:**

**SOP-304**

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**REVISION NUMBER:**

**11**

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**APPROVED BY:**

  
**SECTION MANAGER**

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**QUALITY ASSURANCE OFFICER**

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**EFFECTIVE DATE:**

**09/23/08**

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**DATE OF LAST REVIEW:**

**09/23/08**

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**HERBICIDE - AQUEOUS MATRIX EXTRACTION  
BY EPA Method SW-846 8151A**

**I. SCOPE AND APPLICATION**

1. This SOP describes the extraction of herbicides from water by separatory funnel extraction using SW846 Method 8151A.

**II. SUMMARY**

1. Aqueous samples are extracted with diethyl ether, dried in acidified sodium sulfate, concentrated, and esterified with diazomethane.

**III. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Herbicides can react with alkaline substances and may be lost during the extraction process. Glassware must be acid rinsed and sodium sulfate must be acidified.

**IV. APPARATUS AND MATERIALS**

- Separatory Funnel - 2 Liter with Teflon stopcock
- Beaker - 400 mL
- Fleaker Beaker 1200 mL
- Erlenmeyer flask - 500 mL
- Drying Column (Chromatographic column) - 20 mm I.D. x 300 mm
- Kuderna Danish (KD) flask - 500 mL
- Concentrator tube - 10 mL part of KD setup
- Snyder column - 3 ball, macro
- Plastic connectors - blue to assemble KD
- Water bath - heated, with concentric ring cover, capable of temperature control. The bath should be used in a hood.
- Boiling chips - Teflon
- Vial - 10 mL with Teflon lined lid.
- pH indicator paper - close range (1.0 and 2.0) and (11.0 and 12.0); wide range (1.0 and 12.0)
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, 250 mL, and 100 mL
- Pasteur pipette - length 9" and 5 3/4"

- Pasteur pipette bulb
- Labels - Avery
- Volumetrics - Class A, glass, 1000 mL, 50 mL, 25 mL, a 2 mL graduated pipette, and a 1 mL volumetric pipette.
- Bubbler setup
- Ring stand - 3 prong
- Burette clamp - double
- Rings - large enough to hold KD setup
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator
- Glass manifold - nine positions
- Balance - capable of weighing to 0.1 grams.
- Mortar and pestle

## V. REAGENTS

- Reagent water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.
- Sodium Hydroxide solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl periodically until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-Liter volumetric flask with several rinses of reagent water and dilute to 1 Liter with reagent water. Transfer to a 1000-mL Teflon container.
- Acidified Sodium Sulfate - In a Pyrex glass dish, pour enough sodium sulfate to cover the bottom. Add 10 mL of concentrated sulfuric acid. Add enough ether to cover the sodium sulfate and stir with a glass rod. Let this mixture stand in the hood until all of the ether has evaporated, stirring occasionally. Put into beakers and store in the 150°C oven until ready to use. **Prepare only one tray at a time to minimize the amount of ether. Pull hood sash all the way down when evaporating ether. Keep anything hot or anything that might ignite the ether away from the hood during evaporation.**
- Acidified Glass Wool - Loosely fill a 400-mL beaker with glass wool. Rinse with 1:1 Hydrochloric Acid (HCL), reagent water, acetone, and ether. Dump waste in

the appropriate waste container. Allow the ether to evaporate and store in the 150°C oven until ready to use.

- Cold Sulfuric Acid solution - (1:3), Slowly add 250 mL of H<sub>2</sub>SO<sub>4</sub> (Baker, suitable for trace metal analysis #9673-33) to 750 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use. Store in the refrigerator at 4°C.
- Potassium Hydroxide solution (37%) Weigh 37 grams of KOH into a 400-mL beaker and cover with reagent water. Swirl until all the pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 100-mL volumetric flask with several rinses of reagent water and dilute to 100 mL with reagent water.
- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent)
- Methanol - suitable for use in spectrophotometry and gas chromatography (Omnisolv MX0484-1 or equivalent)
- Acetone - suitable for spectrophotometry and gas chromatography (Omnisolv AX0116-1 or equivalent)
- Hexane - suitable for spectrophotometry and gas chromatography Omnisolv HX0298-1 or equivalent)
- Ether - ultra-resi analyzed for organic residual analysis (Baker #9259-02 or equivalent)
- Iso-Octane - for pesticide residual analysis (Fisher #0-297 or equivalent)
- Dichlorophenyl acetic acid (DCAA) - purchased from Ultra Scientific (PPS-165) @ 1.0 ppm concentration. Add 1 ml per 1000 mLs of aqueous sample.
- Herbicide Spiking Solution & TCLP Herbicide Spike - A spiking solution is prepared by the GC operator. The same solution is for dual purposes. Use 1 mL of 1-100 ppm solution per 1 liter of aqueous sample. Spiking solution is purchased as an acid.
- Herbicide TCLP Spike – 1mL is added per 100 mL volume. Each matrix type must have its own TCLP spike. From the volume provided by Wet Chemistry, remove a 100 mL aliquot into a suitable container with a Teflon lid, and spike according to the required extraction.
- Sodium Chloride - ultra pure crystals for liquid chromatography (Baker #4058-07 or equivalent) purchased in a 12 kg plastic bucket.

- N-methyl-N-nitroso-p-toluenesulfonamide (Diazald) – High purity (Aldrich Chemical Co., or equivalent), see SOP-328.
- Silicic acid,  $\text{H}_2\text{SiO}_5$  – 100-mesh powder, store at 130°C (see SOP-328).

## VI. PROCEDURE

1. All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary using the following information (DO NOT extract samples for which you have no information!):

Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.

Samples requiring RUSH turn around time may be logged in throughout the day that will require your immediate attention. Login personnel will generally communicate this need.

Check with login throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is on-going, extra QC may be avoided by picking up these extractions on the same day.

2. Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

**SLUDGE** - use only 100 mL and dilute to 1000 mL with reagent water.

**TCLP EXTRACT** - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (TCLP spike) is required per client matrix. Dilute to 1000 mL with reagent water.

**BAD MATRIX** – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any, should be made.

3. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.

4. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse with methylene chloride. Label the separatory funnels as follows on your Avery labels: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Extraction date. The Method Blank label should include all lab #s in this set of samples.
5. Remove acidified sodium sulfate from the 150°C oven and allow it to cool.
6. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, and 1g) and adjust for any corrections. Record the readings in the Extraction calibration/temperature logbook. Weigh 125 grams of Sodium Chloride and transfer it to each sample, spike, and method blank.
7. Using the 1000 mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1 liter of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Add 60 mL of Methylene Chloride to the empty bottle.
8. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

Add 1 ml of (DCAA) Herbicide surrogate to each sample, blank and spike with a glass syringe. Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

**NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.**

For the sample in each analytical batch selected for spiking, add 1.0 mL of the Herbicide Spiking solution measured with a glass syringe. Someone must verify that the spike has been added by placing a check mark on each label as it is added.

Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the Herbicide logbook.

9. Shake until all the sodium chloride is dissolved. Add 10 mL of 10N Sodium Hydroxide to each sample, spike and method blank.
10. Using short-range pH paper (11 to 12), check pH of sample by touching pH - sensitive paper to the drop of liquid hanging from the glass stopper. Insure that pH throughout the sample is changed. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH as required to attain the proper pH (>12).
11. Set timer for 1 hour. Allow the sample to set for 1 hour shaking occasionally. This is the hydrolysis step.
12. All glassware from this point forward should be acid rinsed. This includes the fleaker beaker, the 500 mL Erlenmeyer flask, the dry column or funnel, the KD setup and the 15 mL centrifuge tube. Rinse each piece of glassware with Hexane, reagent water, 1:1 HCL, reagent water, acetone and finally with ether.

**NOTE: This step is extremely critical in that if any surface of the glassware that might come into contact with the extract is not rinsed with acid, it could cause the herbicides to be lost. Therefore the glassware should be completely submerged in a 1:1 HCL solution rather than just rinsed.**

13. Using the balance, weigh 20 grams of acidified sodium sulfate into enough 500 mL Erlenmeyer flasks to cover the number of samples, spikes and method blanks that you are extracting. The acidified sodium sulfate is usually hardened into chunks when it comes from the oven. Use a mortar and pestle to grind up the chunks until they have the consistency of sand.
14. When the hydrolysis time has expired, swirl the 60 mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel. In addition add 60 mLs of methylene chloride to the method blank, LCS, and spikes.
15. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon separatory funnels may be placed in the shaker apparatus with the stopcock slightly open. When this apparatus is used, the shake should be for 3 minutes.

**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.**

16. Allow the sample to set for 10 minutes after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 60 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a waste beaker and discard into the Chlorinated Waste Bottle. **Repeat clean-up steps 15-16 two more times.**
17. Adjust the pH to between 1.0 and 2.0. Using a 50 mL cylinder, add 17 mL of 1:3 COLD H<sub>2</sub>SO<sub>4</sub> to all sample, spikes, and blank. Shake to insure that pH throughout the sample is changed. Check pH with short-range pH paper (1.0 to 2.0). Compare the color to the chart on the pH paper. If the color is not within range add more H<sub>2</sub>SO<sub>4</sub> solution as required to attain the proper pH.
18. Using a 250mL-graduated cylinder, transfer 120 mL of ether to each sample, spike and method blank. Shake for two minutes manually or three minutes if by shaker method and allow the sample to set for 10 minutes.
19. Drain the bottom layer (aqueous phase) of each sample into a fleaker beaker. Let stand for one to two minutes. Water and ether will separate again. Drain water layer as before.
20. Drain the remaining layer (ether phase) of each sample into the Erlenmeyer flasks containing acidified sodium sulfate and pour the aqueous phase back into the separatory funnel.
21. Repeat steps 18 through 20 twice using 60mL of ether and combining the extracts in the Erlenmeyer flask.
22. Put a Teflon stopper into the 500mL flask. Cover with aluminum foil and allow the extract to remain in contact with the acidified sodium sulfate for approximately two hours or overnight in the hood.

**NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however the extract may be held in contact with the sodium sulfate (sealed, and under a fume hood) overnight.**

23. In the logbook marked HERBICIDE extractions, enter the Client name, the Lab #, the date extracted, and anything unusual that may have occurred with this sample.

24. DRYING STEP: Dry the sample by either of the following methods:
- 24A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place an acid rinsed drying column into the burette clamp and transfer a small amount of acidified glass wool to the top of it. Tamp it to the bottom with a 10 mL disposable pipette so that it adequately covers the hole at the bottom. Add approximately 10 cm of Acidified Sodium Sulfate to the column. Rinse with 20 to 30 mL of ether and discard this rinse. OR
- 24B. Set up a ring stand with funnels. Place a small amount of acidified glass wool in the bottom of each funnel. Add ~2" acidified sodium sulfate and rinse with 20-30 mL ether. Discard this rinse.
25. Turn on the steam bath and the Turbovap well before they are needed to insure that they are at proper temperature. The setting for the steam bath should be set (between 4 and 5 which yields a temperature around 65 to 85° C) for Herbicide extractions. A higher temperature could cause some of the compounds to be destroyed. Insure that there is enough water in the bath. (Water should cover the coils and be at approximately 1/4 inch below the surface of the overflow device.) The setting for the turbovap is at approximately 35 C.
26. Rinse an acid rinsed Kuderna Danish flask and an acid rinsed concentrator tube with ether and assemble together with a blue clip. Set this in the ring on the ring stand so that it will collect extract as it passes through the drying column/funnel. Pour the entire extract through the column/funnel. Rinse the 500 mL Erlenmeyer flask twice with 10 to 15 mL of ether and add to the drying column/funnel. Finally, rinse the drying column/funnel with 10 to 15 mL of ether. Add a boiling chip.
27. Record the numbers of the Kuderna Danish, and the concentrator tube in the Herbicide logbook.
28. Slip a 3-ball Snyder column into the Kuderna Danish flask and set it on the water bath so that the concentrator tube is immersed in the hot water. The temperature of the bath should be around 85°C. Rinse the 3-ball Snyder with about 1 to 2 mL ether into the KD. As the extract gets hot, the balls will begin to chatter, but the chambers should not flood with condensed solvent. The extract will evaporate in approximately 15 to 20 minutes. When you can no longer see it boiling above the surface of the bath, you need to keep a close vigil on the sample. Take it from the bath momentarily and check the level of extract in the concentrator tube. Don't allow it to quit boiling before placing

- it back into the water. When it reaches approximately 1/2 the length of the tube or 5 mL, remove it from the bath, set it in a ring on the ring stand and allow it to cool. Remove the 3-ball Snyder and rinse down the sides of the flask with 2 to 3 mL with ether. Dry the union with a paper towel to remove excess water and prevent contamination. Disassemble the KD apparatus being careful not to lose any extract from the concentrator tube. Transfer the extract to a Turbovap tube using a 9" Pasteur pipette.
29. Place the Turbovap Tube in the turbovap. Allow to concentrate to just below the 1 mL mark on the tube. Add enough ether to bring up to 1mL mark on TV tube.
  30. Remove sample from the Turbovap and transfer to 12 mL vial using 9" Pasteur pipette. Add 1 mL of Iso-Octane, and 0.5 mL of methanol to each sample.
  31. Esterification should be done as soon after concentration as possible. At this point you will need to coordinate with the GC operator as to when the extracts will be run. It is recommended that herbicides be analyzed as soon as possible after esterification to prevent trans-esterification and other potential reactions that may occur. **See ESTERIFICATION METHOD (SOP -328)**
  32. Add 4 mL of Diazomethane made using SOP-328. Be sure that the diazomethane has a deep yellow color prior to use. Cover the extract with a Teflon-lined screw cap and allow standing for 20 minutes. A persistent yellow color indicates that esterification is complete and excess diazomethane is present.
  33. After 20 mins, bring to final volume with Hexane using Herb dummy vial. Enter the esterification date, the lot number of the diazomethane used and the initials of the person who esterified the sample extracts in the Herbicide logbook. Fill out the labels for the samples and place on appropriate 12 mL vial. Record the vendor and lot number of the methylene chloride, ethyl ether, isooctane, methanol, and hexane used in the sample extract in the logbook.
  34. Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000 mL graduated cylinder and record the sample volume in the Herbicide logbook and the Avery label to the nearest 10 mL.
  34. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract

refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

## VII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for proper disposal of waste from this sample preparation process. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## VIII. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8151A*
2. *USACE EM 200-1-3, 02-2001; Appendix I; Shell for Analytical Chemistry Requirements*
3. *DOD, Quality Systems Manual for Environment*

## IX. HEALTH AND SAFETY

1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
3. MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.
4. Use extreme caution when working with concentrated acids and bases. Use safety glasses, or goggles, gloves and lab coat or apron.
4. Diazomethane is a carcinogen and may explode under certain conditions. See Method 8151 for details in handling diazomethane.

## X. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

**XI. METHOD PERFORMANCE**

Refer to SOP-208 for method performance.

**DEFINITIONS**

°C - degrees centigrade  
COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample  
µg/L - micrograms per liter  
µL - microliter  
µg/ml - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
RL - reporting limit  
SOP - standard operating procedure  
v/v - volume to volume

Refer to SOP-431 for further definitions

**HERBICIDES**

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**NON-AQUEOUS MATRIX**

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**BY SW-846**

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**METHOD 8151A**

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**SOP NUMBER:**

**SOP-310**

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**REVISION NUMBER:**

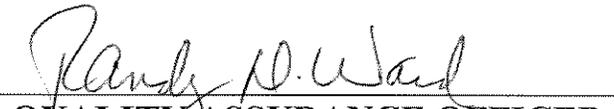
**11**

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**APPROVED BY:**

  
**SECTION MANAGER**

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**QUALITY ASSURANCE OFFICER**

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**EFFECTIVE DATE:**

**09/24/08**

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**DATE OF LAST REVIEW:**

**09/24/08**

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**HERBICIDE - NON-AQUEOUS MATRIX  
Method SW-846 8151A**

**I. SCOPE AND APPLICATION**

1. This SOP describes the extraction of herbicides from soil using SW846 Method 8151A.

**II. SUMMARY**

1. Soil samples are taken through hydrolysis, cleaned up with methylene chloride, extracted with diethyl ether, dried in acidified sodium sulfate, concentrated and esterified with diazomethane.

**III. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Herbicides can react with alkaline substances and may be lost during the extraction process. Glassware must be acid rinsed and sodium sulfate must be acidified.

**IV. APPARATUS AND MATERIALS**

- Balance - Capable of weighing 100 grams to the nearest 0.01 grams
- Teflon Bottles – 250 mL with Teflon screw cap
- Vials - 40 mL and 10 mL glass, with Teflon-lined screw cap
- Syringe - 250  $\mu$ L and 5 mL
- Graduated cylinder - 10 mL, 50 mL, 100 mL, and 250 mL
- Pasteur pipette bulb
- Labels - Avery
- Shaker
- 2000 mL separatory funnels
- 400 mL beakers
- Drying column (Chromatographic column) - 20 mm I.D. x 300 mm.
- Spatula
- Rings to fit Kuderna Danish setup
- Ring stand
- Double burette clamp

**V. REAGENTS**

- Reagent water - Reagent water is NANO PURE WATER gathered in a carboy from source in the instrument lab.
- Potassium Hydroxide solution - (10 N KOH), Weigh 560 g KOH into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all the pellets have dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1 Liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1 liter glass amber jug.
- Potassium Hydroxide solution - (1N KOH), Weigh 56 g KOH into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all the pellets have dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1 Liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1 liter glass amber jug.
- 37% KOH - Weigh 37 grams of KOH into a 400 mL beaker and cover with reagent water. Swirl until all the pellets have dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 100 mL volumetric flask with several rinses of reagent water and dilute to a final volume of 100 mL.
- Methanol - suitable for use in gas chromatography (Omnisolv MX0484-1 or equivalent)
- Ether - ultra resi analyzed for organic residual analysis (Baker #9259-02 or equivalent)
- Iso-Octane - for pesticide residual analysis (Fischer #0-297 or equivalent)
- Hexane - suitable for spectrophotometry and gas chromatography (Omnisolv HX0298-1 or equivalent)
- Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent)
- Dichlorophenyl acetic acid (DCAA) - purchased from Ultra Scientific (PPS-165) @ 1.0 ppm concentration. Add 1 ml of the surrogate DCAA to each non-aqueous sample.
- Herbicide Spiking Solution - A spiking solution containing the herbicide acids is prepared by the GC operator. Add 1 mL of the herbicide spiking solution to the non-aqueous sample.
- Sodium Sulfate ( $\text{Na}_2\text{SO}_4$ ) - Granular, anhydrous, trace pure 10-60 mesh (purchased in plastic bulk containers from Fisher # S415-10S or equivalent) placed in a Pyrex pan and heated at 400°C minimum 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.
- Acidified Sodium Sulfate ( $\text{Na}_2\text{SO}_4$ ) - In a Pyrex glass dish, pour enough sodium sulfate to cover the bottom. Add 10 mL of concentrated sulfuric acid. Add enough ether to cover the sodium sulfate and stir with a glass rod. Let this mixture stand in the hood until all of the

ether has evaporated, stirring occasionally. Put into beakers and store in the 150°C oven until ready to use. **Prepare only one tray at a time to minimize the amount of ether. Pull hood sash all the way down when evaporating ether. Keep anything hot or anything that might ignite the ether away from the hood.**

- Acidified Glass Wool - Silane Treated (purchased from Supelco #2-0410 in a white plastic tub or equivalent). Loosely fill a 400 mL beaker with glass wool. Rinse with 1:1 Hydrochloric Acid (HCL). Reagent water, acetone, and ether. Dump waste in the appropriate waste container. Allow the ether to evaporate and store in the 150°C oven until ready to use.
- Sulfuric Acid solution - (1:3), Slowly add 250 mL of H<sub>2</sub>SO<sub>4</sub> (Baker, suitable for trace metal analysis #9673-33) to 750 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use. Store in the refrigerator at 4°C.

## VI. PROCEDURE

1. All soils have a fourteen-day holding time counted from the hour they are sampled. Determine the samples necessary to extract as follows:

Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.

Samples requiring RUSH turn around time may be logged in throughout the day that will require your immediate attention. Login personnel will generally communicate this need.

Check with login throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

2. Get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix, see your supervisor to find out what dilution, if any, should be made.
3. Get out enough 250 mL Teflon bottles (or 40 mL vials if a 10 gram sample weight is used) to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). Place an Avery label on each 250 mL Teflon bottle or 40 mL vial containing the following information: Lab #, Client name, Type of Analysis, Initial Volume-Final Volume, and the Extraction date. The Method Blank and LCS label should include all lab #s in this set of samples.

4. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trash can.
5. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container**

6. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record the reading in the Extraction Calibration/temperature logbook. Place a 250 mL Teflon bottle (or 40 mL vial if a 10 gram sample weight is used) on the balance and zero it. Using a spatula, transfer approximately 30 grams of sample to the nearest 0.1-gram. Record this amount on your label. Put your label on the side of the 250 mL Teflon bottle (or 40 mL vial). For spiking purposes, weigh up three Teflon bottles with approximately 30 grams of sample each (or three 40 mL vials of a 10 grams each). Pick a sample with a good matrix, one that mixes well, non-oily, etc. The method blank will be an empty extraction container.

**NOTE: Smaller sample weights may be used; reagent/extraction solvent volumes will require adjustment for the size sample used. (Example 10 gram weight then use 1/3 the amount of reagents). Surrogates and standard spike concentrations will remain the same since final sample volumes will not change.**

7. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

Add 1 ml of DCAA to each sample, spike and method blank with a glass syringe. Someone must verify that the surrogate has been added by placing a check mark on the label as the surrogate is added.

**NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.**

For the sample in each analytical batch selected for spiking, add 1 mL of the Herbicide Spiking solution measured with a glass syringe. Someone must verify that the spike has been added by placing a check mark on the label as the spike is added.

Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the extraction logbook.

8. Using a 50 mL graduated cylinder, add 30 mL of 10N KOH to each sample, spike, and method blank. Using a 100 mL cylinder, add 60 mL of Methanol to each sample, spike and method blank. **(Remember to adjust reagents/extraction solvent volumes for the sample weight used.)**
9. Place in shaker, flip switch to slow, and set timer to shake for 30 minutes.
10. In the log book marked Herbicide extractions, enter the Client name, the Lab #, the date extracted, the initial weight and the final volume and anything unusual that may have occurred with this sample.
11. When the timer goes off, remove the samples from the shaker and centrifuge them. Push the "ON" button to turn the centrifuge on. The sample must always be balanced. If necessary, use a dummy bottle with similar weight using reagent water. Set the rpm at 2500 and the temperature at 25°C. Close the lid and be sure to press it down until you hear it lock. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The soil will be packed hard in the bottom of the bottle/vial with the extract on top.
12. Decant the extract into a separatory funnel labeled with the sample #, spike or method blank information.

13. Using a 50 mL graduated cylinder, pour 30 mL of Methanol into each sample, spike and Method Blank. Using a 100 mL graduated cylinder, pour 60 mL of 1.0 N KOH into each sample, spike and method blank. **(Remember to adjust reagents/extraction solvent volumes for the sample weight used.)**
14. Take a glass rod and stir up the packed soil in the bottom of the 250 mL bottle (or 40 mL vial), so that the methanol/ KOH mixture will come in contact with each particle of soil.
15. Place in a shaker, flip switch to slow, and set timer for 15 minutes.
16. Repeat steps 13 through 15 one more time. Combine the extracts.
17. Discard the soil in the trash.
18. Using a 1-L graduated cylinder, add 750 mL of reagent water to each sample, spike and method blank. **(Remember to adjust reagents/extraction solvent volumes for the sample weight used.)**
19. Using a 50 mL graduated cylinder, add 50 mL of methylene chloride to each sample, spike and method blank. Seal and shake the separatory funnel vigorously for 1 minute with periodic venting to release excess pressure. If using shaker method, shake for 2 minutes.

**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.**

20. Allow the sample to set for 10 minutes after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 50 mL into a 250 mL centrifuge bottle. Follow the information given in STEP 10 on the centrifuge; only use the orange holders for the 250 mL Teflon bottles. Use a 9" Pasteur pipette to remove the water layer and return this to the separatory funnel. Discard the remaining solvent into the chlorinated waste-container in the hood.
21. If the layers clearly separate, drain the solvent layer into a waste beaker and discard in the chlorinated waste container in the hood. **Repeat clean-up steps 19-21 two more times.**
22. Acidify to pH <2, using a 200 mL graduated cylinder and adding approximately 60 to 120 mL of cold 1:3 H<sub>2</sub>SO<sub>4</sub>. Stopper and shake to insure that pH throughout the sample is changed. Check the pH with short-range pH paper (1.0 to 2.0). Compare the color to the chart on the pH paper. If the color is not within range, add more H<sub>2</sub>SO<sub>4</sub> solution as required to attain the proper pH. **(Remember to adjust reagents/extraction solvent volumes for the sample weight used.)**

23. All glassware from this point forward should be acid rinsed. This includes the 400 mL beaker, the dry column, the KD setup and the 15 mL centrifuge tube. Rinse each piece with 1:1 HCL, reagent, acetone and finally with ether.
24. Using a 50 mL graduated cylinder, add 50 mL of methylene chloride to each sample, spike, and method blank. Shake for 2 minutes manually or three minutes if by shaker method and allow the sample to set for 10 minutes. Collect the extract in a 400 mL beaker. Extract twice more in the same manner with 50 mL of methylene chloride each time.
25. In the logbook marked Herbicide extractions, enter the vendor and lot number of the methylene chloride used in the sample extract.
26. Turn on the Turbovap and set temp to 35.
27. Dry the sample by either of the following methods:
  - 27A: Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place an acid rinsed drying column into the burette clamp and transfer a small amount of acidified glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Acidified Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse.
  - 27B: Set up a ring stand with funnels. Place a small amount of acidified glass wool in the bottom of each funnel. Add ~2" acidified sodium sulfate and rinse with 20-30 mL methylene chloride. Discard this rinse.
28. Remove any water layer from the extract in the beaker by one of two methods. (1) Remove with a Pasteur pipette by carefully pulling up the water layer on top and not the solvent. Discard this water layer. (2) Sprinkle the smallest amount possible of acidified  $\text{Na}_2\text{SO}_4$  on the top layer until it hardens, separates, and drops to the bottom.
29. Pour sample through funnel into acid washed TV tube. Record the TV tube ID in the logbook. Rinse 400 mL beaker with 10-15 mL ether and pour into TV tube. Rinse Acidified  $\text{Na}_2\text{SO}_4$  with 15-20 mL ether. Allow ether to finish dripping into TV tube.
30. Allow sample to concentrate down completely. When Turbovap signals, the sample should be just below the 1 mL mark on the TV tube. Add 50 mL ether and place back into turbovap for ether exchange.

31. Allow sample to concentrate completely again. When Turbovap signals, the sample should be just below the 1 mL mark on the TV tube. Add just enough ether to bring to 1 mL.
32. Using 9" Pasteur pipette, transfer sample to 12 mL labeled vial.
33. Using the syringes marked Iso-Octane and Methanol, add 1.0 mL of Iso-Octane, and 0.5 mL of Methanol to each sample, spike and method blank. Esterification should be done as soon after concentration as possible. At this point you will need to coordinate with the GC operator as to when the extracts will be run. It is recommended that herbicides be analyzed as soon as possible after esterification to prevent trans-esterification and other potential reactions that may occur.
34. **See SOP-328 ESTERIFICATION METHOD.**
35. Add 4 mL of Diazomethane made using SOP-328. (Be sure that the diazomethane has a deep yellow color prior to use.) Cover the extract with a Teflon-lined screw cap and allow it to set for approximately 20 minutes.
36. Bring to final volume of 10 mL with hexane using the 10 mL Herb dummy vial. Enter the esterification date, the lot number of the diazomethane used, and the initials of the person who esterified the sample extracts in the Herbicide logbook. Print out and complete the labels for the samples. Label each vial with the appropriate label. Record the vendor and lot number of the methylene chloride, ethyl ether, isooctane, methanol, and hexane used in the sample extract.
37. The extract obtained may now be analyzed. Refrigerate at 4°C. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, your initials, and the date and time the samples were placed into the refrigerator.

## VII. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8151A*
2. *USACE EM 200-1-3, 02-2001; Appendix I; Shell for Analytical Chemistry Requirements*
3. *DOD, Quality Systems Manual for Environmental Laboratories, 6-2002*

## VIII. HEALTH AND SAFETY

1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
3. MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.
4. Use extreme caution when working with concentrated acids and bases. Use safety glasses, or goggles, gloves and lab coat or apron.
5. Diazomethane is a carcinogen and may explode under certain conditions. See Method 8151 for details in handling diazomethane.

#### **IX. WASTE MANAGEMENT AND POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for proper disposal of waste from this sample preparation process. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### **X. DOCUMENTATION OF CAPABILITY (DOC)**

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

#### **XI. METHOD PERFORMANCE**

Refer to SOP-208 for method performance.

#### **DEFINITIONS**

°C - degrees centigrade  
COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample

$\mu\text{g/L}$  - micrograms per liter  
 $\mu\text{L}$  - microliter  
 $\mu\text{g/ml}$  - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
RL - reporting limit  
SOP - standard operating procedure  
v/v - volume to volume

Refer to SOP-431 for further definitions

**SOXHLET EXTRACTION -**  

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**BNA AND PEST/PCB**  

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**USING SW846 METHOD 3541**  

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**SOP NUMBER:** SOP-329

**REVISION NUMBER:** 17

**APPROVED BY:**   
**SECTION MANAGER**

  
**QUALITY ASSURANCE OFFICER**

**EFFECTIVE DATE:** 06/22/09

**DATE OF LAST REVIEW:** 06/22/09

**BNA& Pesticide/PCB NON-AQUEOUS MATRIX  
(Soxhlet Extraction)  
USING SW846 METHOD 3541**

**I. SCOPE AND APPLICATION**

1. This SOP describes the extraction of BNAs and pesticides/PCBs from soil, sediment, sludges and waste solids by an automated method (3541).

**II. SUMMARY**

1. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Soxtherm extractor for BNAs or Pesticides/PCBs.. The extracts are then concentrated by either a TurboVap concentrator or by Kuderna- Danish.

**III. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**IV. APPARATUS AND MATERIALS**

- Stainless steel spatula
- Soxtherm automated extractor unit-with 6 position condenser assemblies, internal plumbing, electronic components, stand alone controller unit and a collection tank for spent solvent
- Soxtherm extraction beakers-54 x 130 mm, capacity approximately 125-175 mL
- Suitable thimble (33 mm diameter by 80 to 94 mm length cellulose or equivalent
- Drying Column (Chromatographic column) - 20mm I.D. x 300mm
- Boiling chips - Teflon
- Vial – 2-mL amber with Teflon-lined screw cap
- Vial – 12-mL clear with Teflon-lined screw cap
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Ring stand - 3 prong
- Burette clamp - double
- Rings

- Aluminum foil - heavy duty
- Nitrogen tank - equipped with pressure regulator
- TurboVap Concentrator with 200 ml concentrator tubes
- Balance - capable of weighing to 0.1 grams.
- Aluminum pie pans for mixing samples
- Glass wool- Contaminant free.

## V. REAGENTS

- Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>) - Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher # S415-10S or equivalent) placed in a Pyrex tray and heated at 400 C for a minimum of 4 hrs, removed and then cooled in open air in the extraction lab, placed in a 2.5kg glass amber jug and left at room temperature.
- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).
- Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane) - suitable for spectrophotometry and gas chromatography (Omnisolv #DX0831-1 or equivalent)
- Acetone - suitable for spectrophotometry and gas chromatography (Omnisolv AX0116-1 or equivalent)
- Hexane - suitable for spectrophotometry and gas chromatography (Omnisolv HX0298-1 or equivalent)
- Surrogate/Spike Solutions - Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
  1. **BNA Surrogate (100 ug/ml)** - The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low level PAHs use 1.0 ml of 1.0 µg/mL BN Surrogate spiking solution.)**
  2. **BNA Spiking Solution (100 ug/ml)** - The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample.**(For low level PAHs use 1.0 ml of 1.0 µg/mL PAH spiking solution.) The BNA Spiking solutions contains all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
  3. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl)** Warm and sonicate - Surrogate solution is prepared in acetone by making a cut on stock purchased from a

- reputable vendor. 0.5 mL at 0.5 µg/mL of this solution is added per 15g of non-aqueous sample.
4. **PCB Spiking Solution-** Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0 ug/ml. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5 mL per 15.0 grams of non-aqueous sample.
  5. **Pesticide Spiking Solution** - A spiking solution is prepared at 1.0 ug/ml. Use 0.5 mL per 15 grams of non-aqueous sample.

## VI. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

1. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a teflon-lined cap.
2. Samples are preserved by cooling to 4° C.
3. Holding time is 14 days from collection date to extraction.

## VII. PROCEDURE

### Extraction Procedure

1. All soils have a 14 day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
2. Each day a backlog report is pulled in ELEMENT and samples are extracted based on priority.
  - Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
  - Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
2. Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a soxhlet extraction is truly necessary.
3. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes or LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples).

4. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
5. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container**

6. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15 gram sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.
7. Add ~ 20 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, simply weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
8. Transfer the sample and its label to a soxtherm beaker, which has been prepared using a glass rod to place glass wool in and over the hole at the bottom side.

9. Add 120 mL of solvent, methylene chloride for BNAs or hexane for pesticides/PCBs, to the Soxtherm beaker for each sample, method blank, and spike - add boiling chips.
10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by placing a check mark on the label as it is added.

NOTE: Surrogate and spike should be added just prior to setting on the Soxtherm.

Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike.

For the BNA sample in each analytical batch selected for spiking, use the 1.0-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. . **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**

For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:

**Pesticide and PCB** - set up two LCS's – one for Pesticide getting a MIX A&B spike and one for PCB which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

**Pesticide only** – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

**PCB only** - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na<sub>2</sub>SO<sub>4</sub>.

11. **Automated Soxhlet:** The *Soxtherm* will extract any number of samples up to six per run. Generally, we set up to complete four sets of six a day or/as sample load permits with required QC. Each extraction cycle takes 1-2 hours. Reference *O.I. Analytical Operator's Manual* for programming and making adjustments to extraction cycle settings.
  - The extraction thimbles are placed into the respective extraction thimble holders(the open end of the thimble is nearly flush with the upper edge of the metal ring. Transfer the sample into the extraction thimble. The appropriate spiking solution should be added at this point (surrogates, MS/MSD or LCS etc...). (Placing a small piece of glass wool sufficient to cover the diameter of the thimble will help hold the sample mixture in the thimble.)
  - For loading samples on the *Soxtherm*, the condensers should be in their raised position. If Viton gaskets are to be used, install them in the extraction beakers so that a good seal will form between

the extraction beaker and the Teflon extraction cylinder. Install the extraction beakers by depressing the holding clamp and carefully sliding the beaker onto or off the bottom of the Teflon fitting that is situated below the glass condenser. After the extraction beakers containing the samples have been loaded on the *Soxtherm*, push all beakers against the rear limit stop of the hot plate.

- The *Soxtherm* is now ready to run with one of the programs listed below (**remember to check the sight glass on the front of the Extraction Unit to see if the solvent collection tank is near full and should be drained**).

#### Extraction Program

Temperature	= 165°C (Maximum temperature 200°C)
Boiling Time	= 30 minutes
Solution Reduction A	= 5x15 ml
Extraction Time	= 30 minutes
Solution Reduction B	= 0 minutes
Solution Cooling	= 0 minutes
Solution Reduction Interval	= 2.5 minutes
Solution Reduction Pulse	= 2 seconds

- At the end of Solution Cooling time the condensers and extraction beaker assemblies are automatically raised off the heating block and the process completion message appears on the display. Remove the beakers to a hood and continue with the process as listed below.

13. Labels for extract vials are printed from ELEMENT after extraction information is entered.

14. Dry the sample by either of the following methods:

14A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride or hexane and discard this rinse into the appropriate waste container (make certain that methylene chloride waste goes in the chlorinated solvent waste container in the hood).

14B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it. Add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride or hexane for Pest/PCBs. Discard this rinse.

15. TURBO-VAP CONCENTRATION

- A. Turn on the TurboVap and set the temperature at 40°C -50°C. This temperature is necessary to evaporate the acetone. Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve.
- B. Rinse a Turbo-Vap tube and arrange it underneath a rinsed, packed drying column/funnel. Pour the extract through the column/funnel so that it will collect in the tube. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. Therefore the process cannot be completed in one step. Pour enough of the extract from the 400 mL beaker to reach the 200 mL level mark on the Turbo-Vap tube. To help prevent cross contamination, cover the turbovap tube with aluminum foil and punch a small hole in the top of it. A portion of the extract will remain in the beaker. This should be set aside with the funnel used to dry the first portion of the sample placed in the mouth of the beaker.
- C. Record the Turbo-Vap tube number, the vendor and lot number of hexane used in the appropriate extraction logbook.
- D. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- E. When the extract has been concentrated to ~1.0 mL, remove it from the Turbo-Vap and find the remaining extract. Reuse the original funnel and pour the remaining extract through it so that it drains into the turbovap tube. Rinse the 400 mL beaker twice with 10 -20 mLs of methylene chloride or hexane for Pest/PCBs transferring the rinse to the funnel each time. Finally rinse the funnel with ~ 10 mLs of solvent. Place a piece of aluminum foil over the tube and with a sharpie mark an "X" on the aluminum foil. Remove the tube to a metal holder. Concentrate as before.
- F. When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL, remove the tube from the bath. The extracts are ready to be transferred into their appropriate vials.
- **For Pest/PCB Completion:** Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" pasteur pipette to draw up the sample and transfer it to the 10 mL vial. Be careful not to spill any sample during the transfer because one drop represents 5% of the sample and is a critical loss.
  - Fill a 10 mL graduated cylinder with 9.5 mL of hexane. Add 1/2 of the hexane to the tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 10 mL vial. Add the remaining hexane and repeat this rinsing process. Cover the extract with a Teflon-sealed screw cap.
  - **BNA Completion:** When the volume has been reduced to 1-0.5(~0.4)mL, remove the tube from the Turbo-Vap and place in a metal holder.

- Use a 9" Pasteur pipette to transfer the extract to a 2 mL amber vial. **THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!!** A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.20 mL of methylene chloride into a 0.50 mL syringe and add ~ half of this aliquot to the tube. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2 mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial.
- Compare this volume to a 2 mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume.

Compare the volume to a 2mL dummy vial containing 0.5 mL of solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

NOTE: If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Refer to SOPs 307 to 309 and 330, 331, 333 for further cleanup information. Discuss cleanup possibilities with the analyst and/or your supervisor.

The extract obtained above may now be analyzed. Refrigerate at 4°C or carry directly to the analyst. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, your initials, and the date and time the samples were placed into the refrigerator.

Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

## VIII. DOCUMENTATION OF CAPABILITY ( DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

**IX. WASTE MANAGEMENT AND POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

**X. METHOD PERFORMANCE**

Refer to SOP-201 and SOP-211 for method performance.

**XI. REFERENCES**

1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 3541.

**DEFINITIONS**

BNA - Base/ Neutral/Acid  
°C - degrees centigrade  
COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample  
µg/L - micrograms per liter  
µL - microliter  
µg/ml - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
PAH - polynuclear aromatic hydrocarbons  
PCBs - polychlorinated biphenyls  
Pest - pesticides  
RL - reporting limit  
SOP - standard operating procedure  
TCMX - tetrachloro-m-xylene  
v/v - volume to volume

Refer to SOP-431 for further definitions

**LABORATORY SAMPLE RECEIVING,  
LOG IN AND STORAGE  
STANDARD OPERATING PROCEDURES**

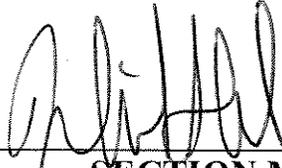
**SOP NUMBER:**

**SOP-404**

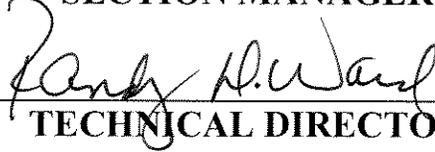
**REVISION NUMBER:**

**13**

**APPROVED BY:**



**SECTION MANAGER**



**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:**

**06/29/09**

**DATE OF LAST REVIEW :**

**06/29/09**

## LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

### SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the “Sample Receiving” section of this document.

### **I. Sample Receiving**

A. Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our

coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment II]* is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.
- B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."
1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.
  2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. **THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.**
- C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.
- D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign then list the date and time received on the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined for

leakage, breakage etc., the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.
  2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.
  3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.
- E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.
1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.
  2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

**The only exceptions to the 6°C rule are:**

- a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO<sub>3</sub> to a pH of  $\leq 2$ . *Keep in mind that non-aqueous sample for Metals must be cooled.*
  - b. Samples for Fluoride, Chloride and Bromide.
  - c. Waste/Product samples for all parameters.
  - d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.
  - e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received On Ice" (ROI).
  - f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.
- F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:
1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.
  - a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.
  - b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.
3. Samples requiring rush turnaround.
  - a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.
  - b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.
- G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.
  1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.
  2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.
- H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.
  2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.
- I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

## II. Sample Log In

- A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.
- B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.
1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)
  2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.
  3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).
- C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. *When samples are unpacked they must be placed in cold storage within two hours even if they have not been through the entire log in procedure.* All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)
2. The sample descriptions on the bottle should match those on the chain of custody. (See section IH)
3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO<sub>3</sub> used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.
  - a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.
  - b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather than a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

- c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH, Electrometric.]
4. The following guidelines must be followed to check pH preservation:
- a. Water samples for Cyanide analyses must be preserved to a pH of  $>12$  with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to  $\geq 12.0$  unless project/client specific requirements are to contact the client first.
  - b. Water samples for Metals analyses must be preserved to a pH of  $\leq 2.0$  with HNO<sub>3</sub> upon collection. If the pH of these samples is between 2.0-3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to  $\leq 2.0$ . unless project/client specific requirements are to contact the client first.
  - c. Samples requiring analyses which are preserved with H<sub>2</sub>SO<sub>4</sub> (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted  $\leq 2.0$  unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH  $>9$ .
  - d. If a sample is not properly preserved, log-in personnel must either do the following:
    - To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.
    - For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.
    - All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be CAR generated and the client notified to see if the lab is to proceed with analysis.

- e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).
  - f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.
5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.
  6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (0.2g) when chlorine is known to be, or suspected to be present.
  7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.
  8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.
  9. Be aware of holding time requirements. (See section 1D)

- D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VIII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:
1. Client and Log #s
  2. Date/Time Unpacked
  3. Logged In/Numbered By (Initials)
  4. 2nd Checked By (Initials)
  5. Date/Time Placed in Cold Storage
  6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
  7. Disposed of By/Date
  8. Method of Disposal
- E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

### III. Sample Storage

- A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***
1. The Hobart refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***
  2. Walk In Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.
- B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:
1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled *Soil Movement Regulations [Attachment VIII]* is posted in the log-in room.
  2. All soils taken from foreign sources, US Territories and Hawaii.

**NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.**

- C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:
1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)
  2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.
- D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year (**digital thermometers quarterly**) and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

- E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the *Sample Custody Form [Attachment IX]* which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]
- F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.
- G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.
- H. A temperature maintenance record book is kept for each refrigerator.
- I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

#### IV. Laboratory Information Management System (LIMS)

- A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. **Client:**

Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. **Projects:**

Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

- a. Internal -- Empirical Laboratories projects;
- b. External -- direct laboratory clients.

3. **Comments:**

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. **Received By:**

Enter the name of the person who received the samples.

5. **Logged In By:**

Enter the name of the person who logged in the samples.

6. **Received:**

Enter the date and time received separated by a space and using military time.

Example: 08/02/2008 08:30

7. **Project/Package Date Due:**

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. **Shipping Containers:**

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. **COC Number:**

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By:***

Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

*After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.*

11. ***Sample Name:***

- a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.
- b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.
- c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # \_\_\_\_". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:00 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, a CAR must be generated. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with

regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***pH (Container Preservative):***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

17. ***Comments:***

Enter any information that is applicable at the sample level.

18. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

19. ***Work Analyses:***

Select all parameters requested for the sample from this list.

- a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

- b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.
- c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

20. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

21. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

22. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

- a. A 2-digit numeral of the year. Example (0811248-06)
- b. A 2-digit numeral of the month. Example (0811248-06)
- c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)
- d. A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

23. ***Add/Edit/Copy:***

Use these selections to add more samples to the work order, or to change existing information prior to label printing.

*Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.*

- B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

#### V. Daily Follow Up for Sample Receiving/Log In

- A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.
- B. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).
- C. Complete any required CARs.
- D. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):
1. The original chains of custody and yellow original or copy of each. The CRF will accompany the COC for the project.
  2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.
  3. All CARs.
  4. LIMS sample receiving logs.
  5. Copies of any RUSH sheets which have been distributed
  6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.
- E. All the above information from the day will be reviewed as soon as possible.
1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.
  2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a *Sample Log Change Form [Attachment XIII]* through email distribution. A *Sample Log Change Form* by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.
- F. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:
1. Copies of the LIMS receiving reports to necessary laboratory personnel.
  2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.
  3. Finalized/approved CARs must be sent to the:
    - a. Organic Manager
    - b. Inorganic Manager
    - c. Laboratory Manager
    - d. Laboratory Director {optional}
    - e. Quality Assurance Officer
    - f. Administrative Assistant
    - g. Client {optional}
  4. Copies of any project/sample specific information to the Section Manager and analysts.
- G. Information will be filed as follows:
1. Chains of custody:

- a. Original (white copy) is returned to the customer with the final report along with the CRF.
  - b. Pink copies should be retained by the sampler.
2. CARs
- a. CARs can be found at V:\LAB\log-in\login (year)\logcar (year).
3. Sample Change Forms and RUSH Sheets
- a. Sample Change Forms are distributed by email.
  - b. RUSH Sheets are found at V:\LAB\login\Rushsheets
4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

## VI. Miscellaneous

- A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.
- B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.
- C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.
- D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as *Attachment XIV*.
- E. A *Telephone Conversation Log [Attachment XV]* may be required to document information and may be attached to or used as a CAR.

- F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

<b>Log Book ID</b>	<b>Log Book Description</b>
LI014	Trip Blank Prep Log Book
LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI013	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

**Attachments to SOP 404**

II	Chain of Custody Record
III	Corrective Action Report for Sample Receiving/Log In
IV	Cooler Receipt Form
V	List of Short Holding Time (Immediate-72 hrs.) Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the US.
IX	Laboratory Sample Custody Form for Walk In Refrigerator
X	Container Codes for the LIMS
XI	Routine NPDES Clients
XII	RUSH Sheet
XIII	Sample Log Change Form (Green Sheet)
XIV	Flow Chart, Laboratory Sample Tracking System

*[Attachments I and VI were removed during the editing process and not added to the SOP.]*



**EMPIRICAL LABORATORIES**

**CORRECTIVE ACTION REPORT FOR SAMPLE RECEIVING/LOG-IN**

**Date Completed:**

**Form Completed By:**

**Date Samples Received:**

**Parameter(s):**

**Client/Job #:**

**Samples:**

---

**Problem(s):**

**Action Taken:**

**Action Taken to Prevent Reoccurrence of this Problem:**

**Approval of Section Leader:**

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**Distributed to:**



## Short Holding Time Parameters

(Immediate-72 hours)

Parameter	Holding Time
pH	Immediate <sup>a</sup>
Sulfite	Immediate <sup>a</sup>
Temperature	Immediate <sup>a</sup>
Residual Chlorine	Immediate <sup>a</sup>
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) <i>Drinking Water only</i>	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours <sup>b</sup>

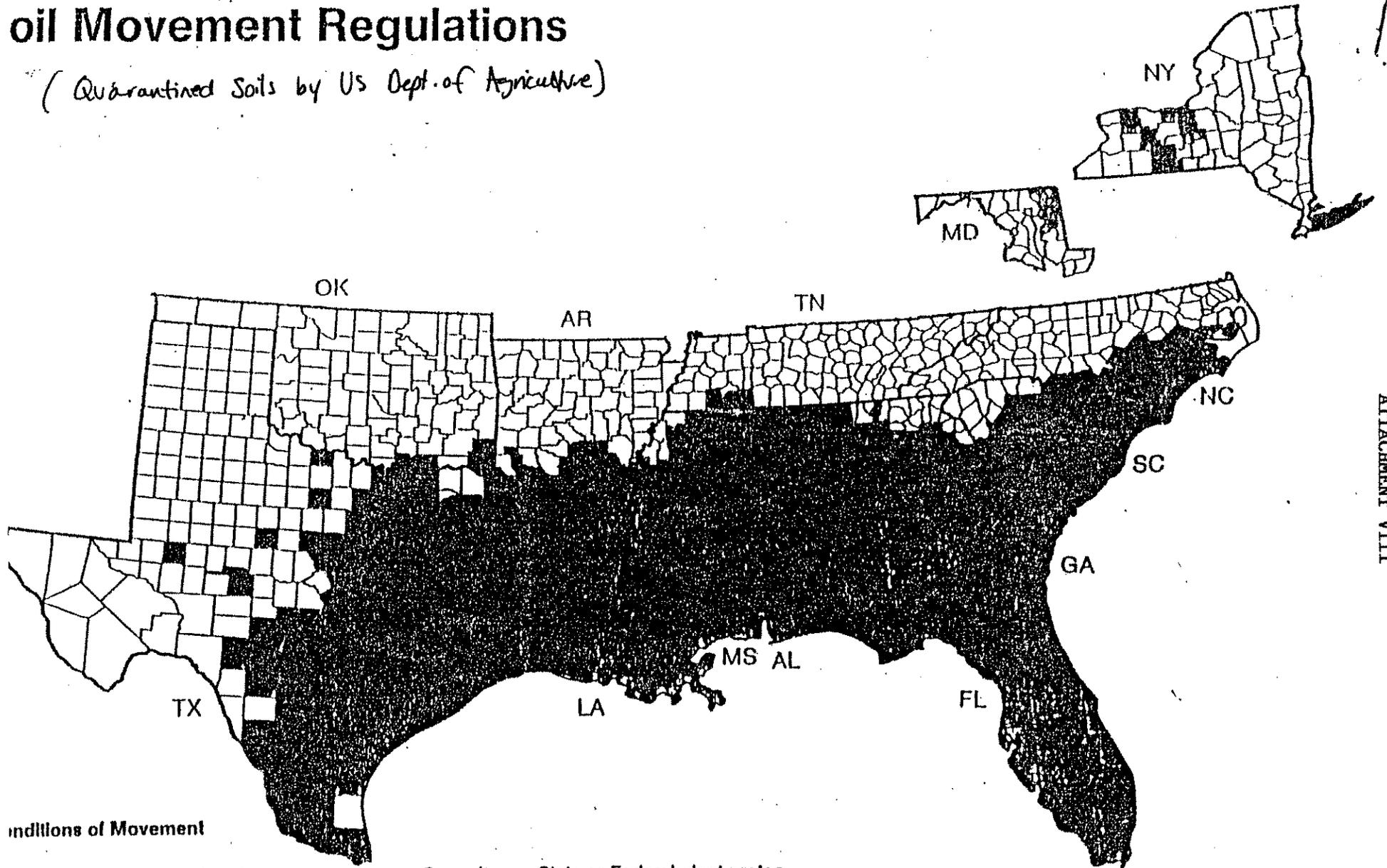
<sup>a</sup> Immediate generally means within 15 minutes of sample collection.

<sup>b</sup> This is an internal holding time. The method does not specify a holding time.



# oil Movement Regulations

(Quarantined Soils by US Dept. of Agriculture)



ATTACHMENT VIII

## Restrictions of Movement

Restrictions are imposed on the movement of regulated articles from a regulated area as follows:  
From red areas into or through white areas. Movement within red areas may be regulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

■ Regulated Area



<b>Preservatives</b>		<b>Types of Container</b>	
<b>NI</b>	<i>HNO3</i>	<b>A</b>	<i>1 LITER - PLASTIC</i>
<b>NF</b>	<i>HNO3 (Filtered)</i>	<b>B</b>	<i>500 mL - PLASTIC</i>
<b>SU</b>	<i>H2SO4</i>	<b>C</b>	<i>250 mL - PLASTIC</i>
<b>SH</b>	<i>NaOH</i>	<b>D</b>	<i>120 mL - PLASTIC</i>
<b>ZN</b>	<i>ZnAC / NaOH</i>	<b>EN</b>	<i>ENCORE PAK</i>
<b>HY</b>	<i>HCl</i>	<b>F</b>	<i>1 LITER - GLASS CLEAR WIDE MOUTH</i>
		<b>G</b>	<i>1 LITER - GLASS CLEAR BOSTON ROUND</i>
		<b>H</b>	<i>1 LITER - GLASS AMBER</i>
		<b>I</b>	<i>250 ml. - AMBER</i>
		<b>J</b>	<i>VOA VIALS - (40 ml.)</i>
		<b>K</b>	<i>500 ml. - (16 oz)</i>
		<b>L</b>	<i>250 ml. - (8 oz)</i>
		<b>M</b>	<i>125 ml. - (4 oz)</i>
		<b>N</b>	<i>60 ml. - (2 oz)</i>
		<b>O</b>	<i>OTHER</i>
		<b>P</b>	<i>PLASTIC BAG -1 Gallon</i>

ROUTINE NPDES CLIENTS (Page 1 of 2)

ALCAN Ingot and Recycling  
Amoco Oil  
Armstrong (Pirelli)  
Atochem-Carrollton, KY  
Auburn Hosiery Mill  
Autostyle

Bando Manufacturing  
Bowers Ink  
Bowling Green Municipalities (City of)  
BP Oil  
Bremner, Inc.  
Brentwood, City of  
Brown Printing Central

Burgill Steel and Wire  
Clarksville Products

Dupont  
Eaton and Olsen  
Emhart Pop Rivets

Fleet Design  
Franklin, City of

Gatlinburg

Hennessey Co. (Coats)  
H.I.S. Laundry  
H.K. Bell (City of Hopkinsville Landfill Monthly Monitoring)  
Hoover

International Paper  
J. S. Technos

Ken Koat  
King Industries

Lannom Tannery  
Leonard Plating

ROUTINE NPDES CLIENTS (Page 2 of 2)

al Plate, Inc.  
Morflex, Inc.

Nashville Wire  
Norandal USA Inc.

Oak Ridge, City of

Plymouth Tube  
Prime Colorants

RMI

Shared Hospital Services  
Snap On Tools  
Springfield, City of  
Special Metals  
Steel Industries

Tennessee Dickel Distillery  
Tulahoma, City of

UCAR, Clarksville

Valmore Leather

Westvaco (Mayfield Creek Up/Down)  
Woodbury

Revised 9/10/96



SAMPLE LOG CHANGE FORM

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DATE:

TO:

SAMPLE #(S):

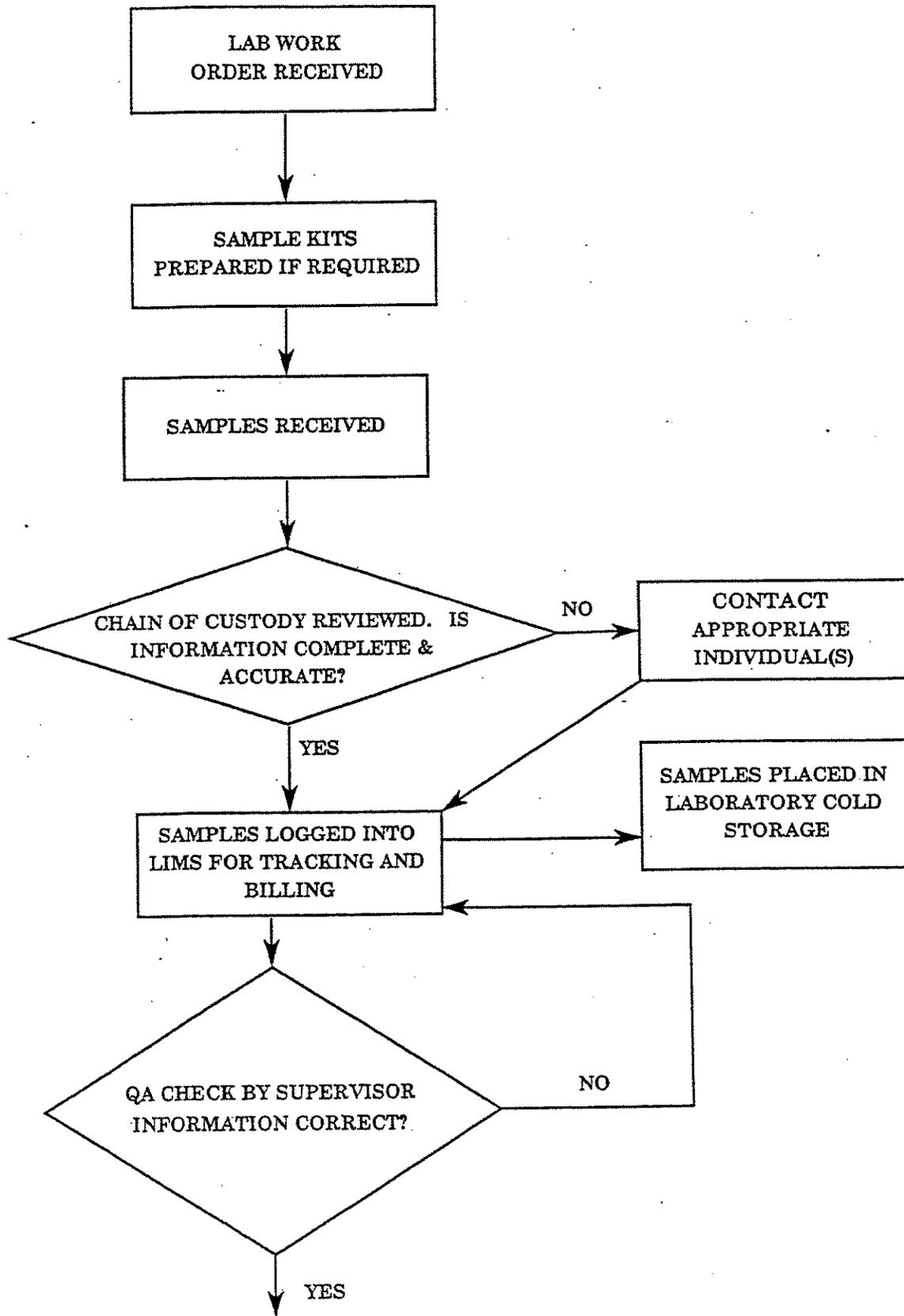
CLIENT:

---

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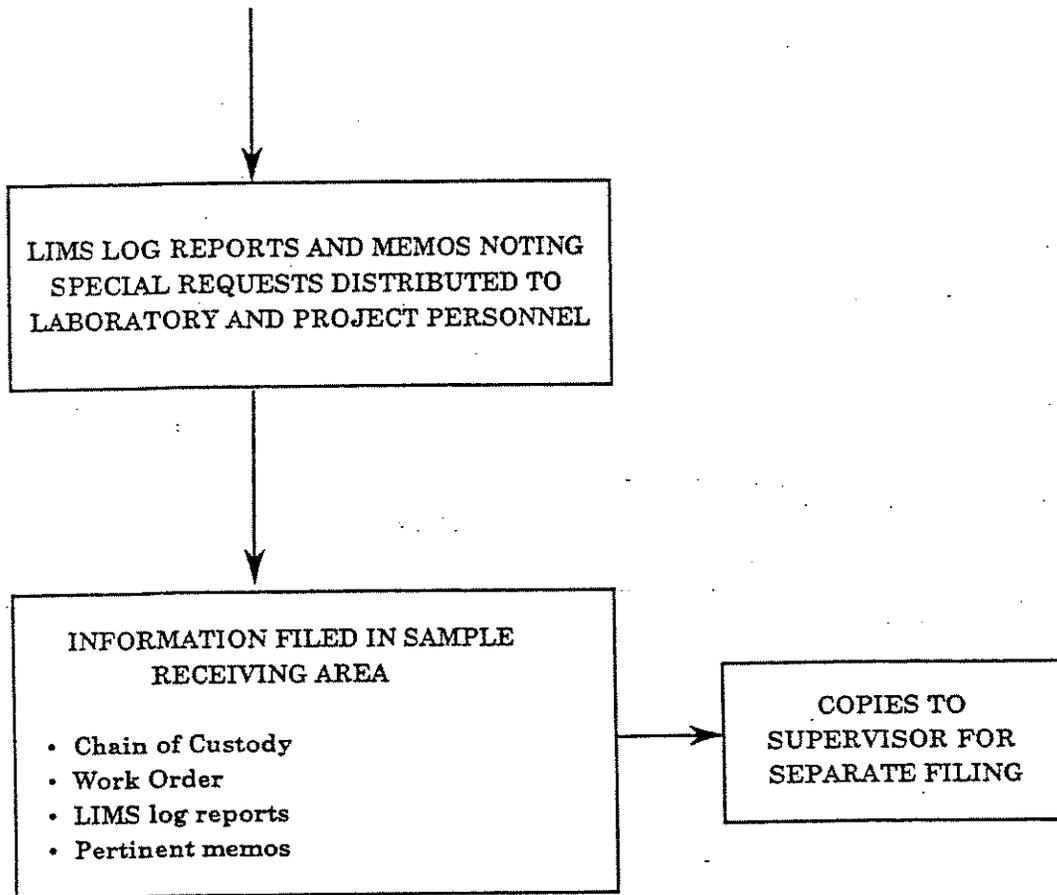
Changes:

LABORATORY SAMPLE TRACKING SYSTEM  
SAMPLE RECEIVING



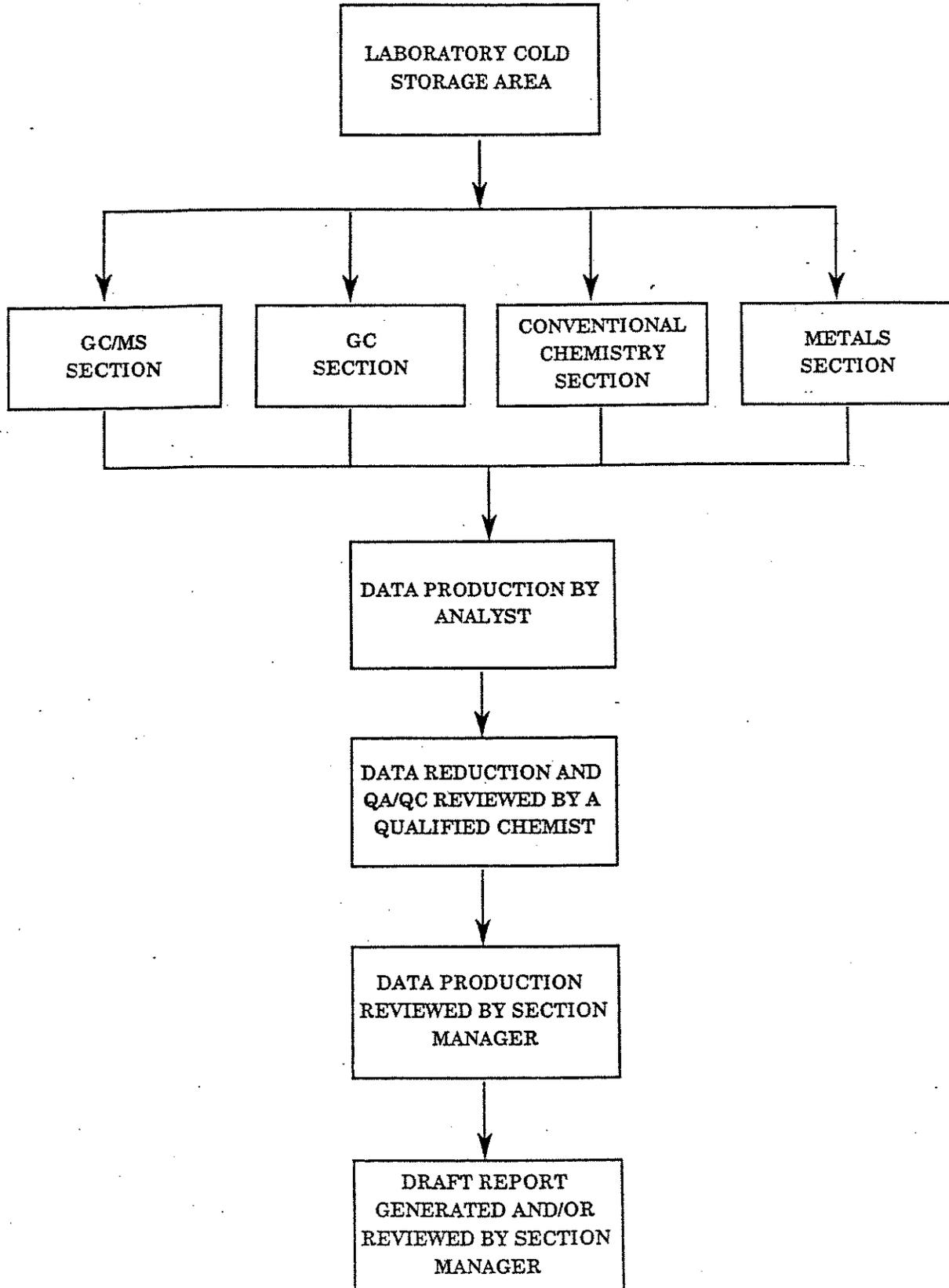
ATTACHMENT XIV (Continued)

LABORATORY SAMPLE TRACKING SYSTEM  
SAMPLE RECEIVING (continued)

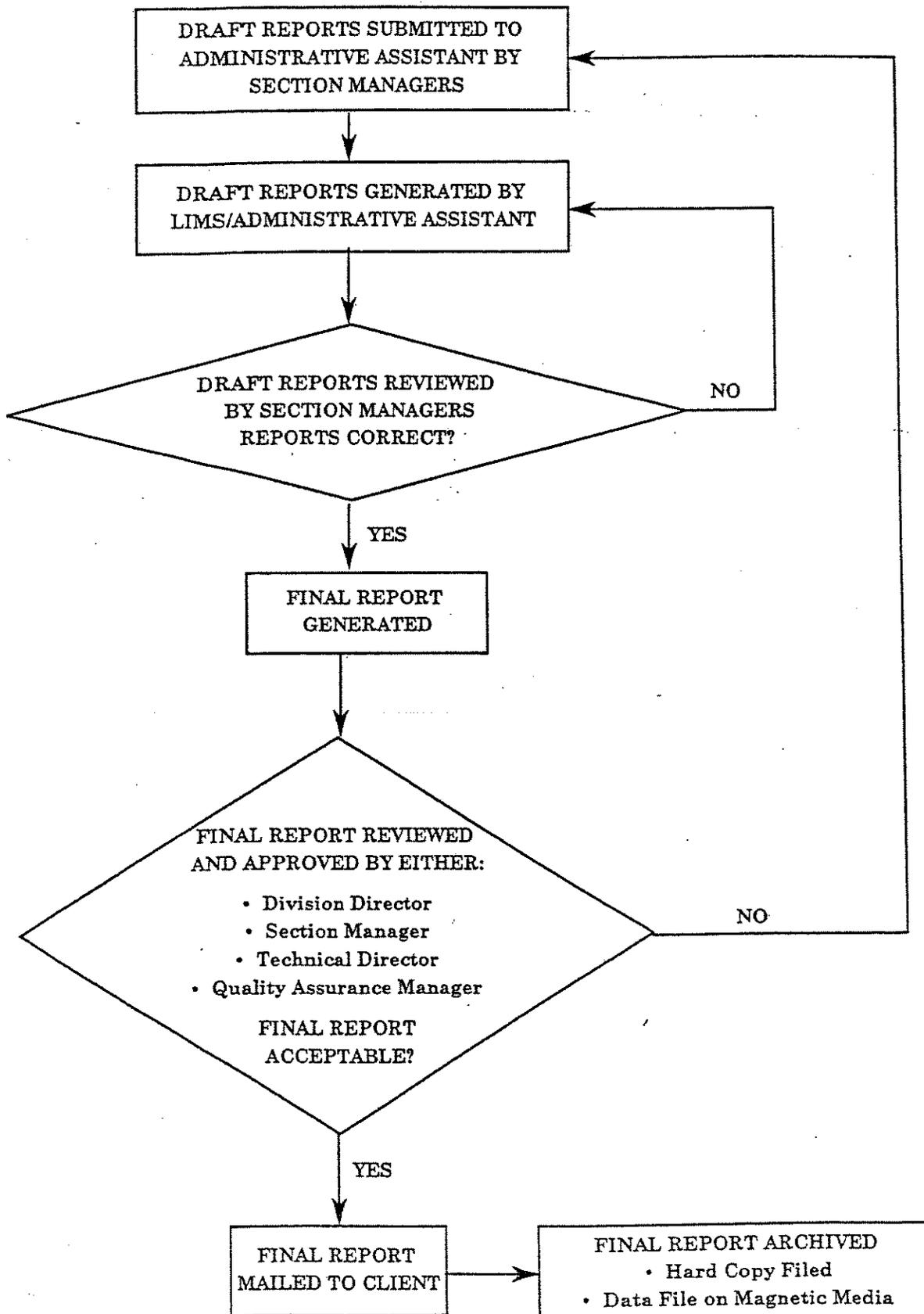


ATTACHMENT XIV (Continued)

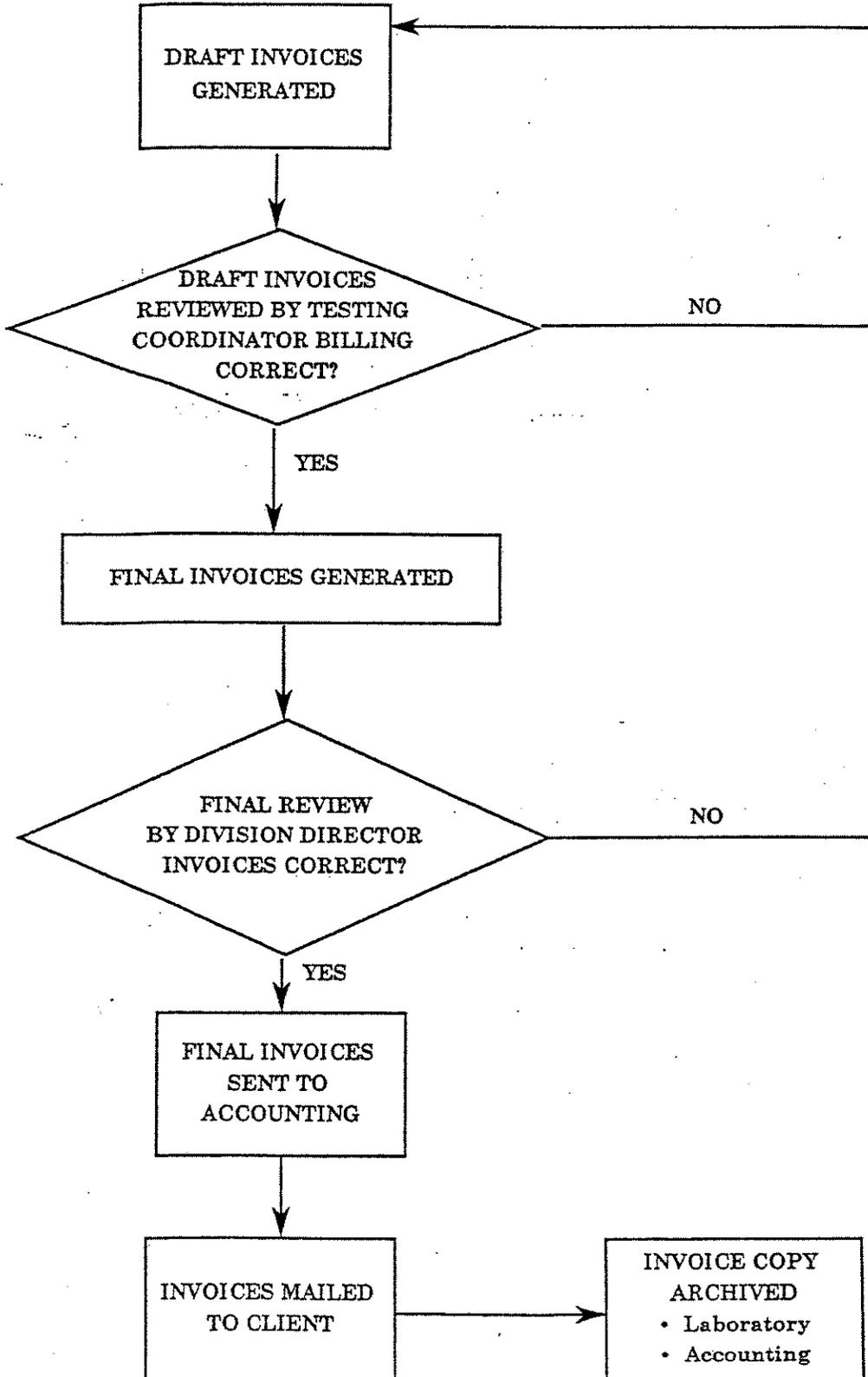
LABORATORY SAMPLE TRACKING SYSTEM  
DATA PRODUCTION AND REVIEW



LABORATORY SAMPLE TRACKING SYSTEM  
DRAFT AND FINAL REPORT



LABORATORY SAMPLE TRACKING SYSTEM  
INVOICING



# DISTRIBUTION/TRAINING LOG

**LABORATORY SAMPLE RECEIVING,  
LOGIN AND STORAGE  
STANDARD OPERATING PROCEDURES**

**SOP NUMBER:**

**SOP-404**

**REVISION NUMBER:**

**13**

**RECEIVED BY/DATE:**

W. Schwab

*W. Schwab* *WS*

F. Rivers

*FR* *FR*

~~R. Townsend~~

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

**TRAINED BY:**

*Landy D. Ward*

**EFFECTIVE DATE:**

**06/29/09**

**PLEASE COLLECT OLD SOPs AND RETURN WITH  
SIGNED FORM TO QAO**

**ANALYTICAL  
LABORATORY WASTE  
DISPOSAL**

---

**SOP NUMBER:**

**SOP-405**

---

**REVISION NUMBER:**

**5**

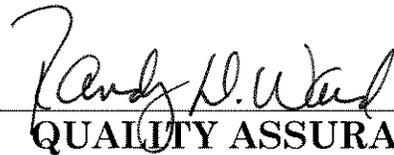
---

**APPROVED BY:**



**LAB DIRECTOR**

---



**QUALITY ASSURANCE  
OFFICER**

---

**EFFECTIVE DATE:**

**06/23/09**

---

**DATE OF LAST REVIEW:**

**06/23/09**

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## **Analytical Laboratory Waste Disposal Standard Operating Procedure**

### **I. SCOPE AND APPLICATION:**

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

#### **A. Wastes can be broken down into the following categories:**

1. Unused portions of actual samples received from outside clients.
  - a. Unused aliquots of completed water samples.
  - b. Unused aliquots of completed non-aqueous samples.
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

### **II. SUMMARY OF PROCEDURES:**

#### **A. There are four options for disposing of unused sample portions:**

1. Return completed samples and any generated waste from these samples to the client.
2. Throw the sample away after confirming that it is non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

**B. There are two options for disposing of laboratory generated waste:**

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

**For example: Solvent waste must be stored in the vented flammable cabinet.**

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

**III. EQUIPMENT/APPARATUS:**

**A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).**

**B. USDOT approved drums for storing and shipping hazardous waste.**

**C. Fume hood vented outside the building.**

**D. Flammable storage cabinet which is vented to the outside**

**IV. PROCEDURE**

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

**A. Disposal of completed aqueous samples:**

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.
  - a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.
  - b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.
  - c. **be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**
  - d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.
  - e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.
  - f. All samples disposed of in this manner must be documented in the bound disposal log.
2. If water samples are hazardous (known or suspected), one of the following steps must be taken.
  - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**
  - b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
  - c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

## **B. Disposal of completed non-aqueous samples:**

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for two hours.

b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this manner must be documented in the bound disposal logbook with the following information:**

a. Client

b. Sample #s

- c. Date(s) treated
- d. Treatment method used

### C. Disposal of laboratory generated waste:

Generated waste is stored outside the building, inside the caged fence until a waste pick up occurs. This area must be maintained properly.

#### 1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

##### a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, (**>2% by volume**) and dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**
- Dilute acid waste (**≤2% by volume or less**) are neutralized using concentrated amounts of sodium hydroxide and poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated along with the amount of sodium hydroxide used to neutralize the acid waste, is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

##### b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed

below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards (**>2% by volume**) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ....etc...) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

**\*\*Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type and quantity of solvent is entered into the *'Organic Solvent Waste Logbook'* located on the shelf next to the drums.**

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located in the GC/HPLC Laboratory.

**PCB Box** – all samples/standards

**Pesticide Box** – all samples/standards

**Herbicide Box** – all samples/standards

**8330 Box** – all samples/standards

**Methylene Chloride Box**- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial. **Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

**Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

**Waste from the instrument** - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain (pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and disposed of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

**Standards - Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company.**

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.
- Hazardous or product samples are returned to the client.

#### **D. Consolidation of satellite waste for contractor disposal:**

In conjunction with the Safety Officer, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence (c. *Organic Extraction Laboratory Area*, \* **Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.
2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.
4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

**It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.**

# DISTRIBUTION/TRAINING LOG

## ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER:

SOP-405

REVISION NUMBER:

5

RECEIVED BY/DATE:

W. Schwab	<i>W. Schwab</i>	
R. Townsend	<i>Russell Townsend</i>	RET
F. Rivers	<i>F. Rivers</i>	FK
J. Holliman	<i>J. Holliman</i>	J.H.
B. DeVille	<i>Betty DeVille</i>	
A. Monteiro	<i>A. Monteiro</i>	
B. Richard	<i>B. Richard</i>	

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY:

*Randy H. Ward*

EFFECTIVE DATE:

06/23/09

**PLEASE COLLECT OLD SOPs AND RETURN WITH  
SIGNED FORM TO QAO**

**STANDARD OPERATING  
PROCEDURE (SOP) FOR  
LABORATORY SAMPLE  
STORAGE, SECURE AREAS  
AND SAMPLE CUSTODY**

---

**SOP NUMBER:**

**SOP-410**

---

**REVISION NUMBER:**

**6**

---

**APPROVED BY:**



**SECTION MANAGER**



**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:**

**09/08/08**

---

**DATE OF LAST REVIEW:**

**09/08/08**

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**STANDARD OPERATING PROCEDURE (SOP) FOR  
LABORATORY SAMPLE STORAGE, SECURE AREAS  
AND SAMPLE CUSTODY**

Empirical Laboratories, LLC is located on the fifth floor of a building which is locked and monitored by a guard after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and the elevators are security coded (i.e. a code must be entered in order to get the elevator to open on the fifth floor.) All stairway doors are locked and only Empirical Laboratories, LLC personnel have a key to the fifth floor stairway door. The doors to the lab in the hallway have a key code. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Shipping/Sample Receiving (SR) area on the fifth floor. The laboratory is located close to Federal Express (FedEx) distribution station, therefore we pick up our coolers at the FedEx location on Saturdays and transport them directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client. The SR personnel must not leave any packages/cooler without authorized receipt from laboratory personnel. Samples must be accompanied by some type of chain of custody record. Sample receiving personnel sign, and list the date and time received on the chain of custody. The time received must reflect the actual time or validation date and time of receipt for the samples although they may be placed in cold storage and logged into the system at a later time. The method of delivery is listed on the cooler receipt form(CRF). The tracking # (if available) is attached to the chain of custody.

Once sample containers have been assigned a laboratory ID number, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A Sample Receiving Custody and Disposal Form (attached) must be completed each day. Samples should not leave the log-in area until this has been completed. A copy of this form must be given to the Testing Coordinator at the end of the day. The original is to remain in Sample Receiving until the samples are disposed. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- Client and Log #s
- Date/Time Unpacked
- Logged In/Numbered By (Initials)
- 2<sup>nd</sup> Checked By (Initials)
- Date/Time Placed in Cold Storage
- Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- Disposed of By/Date
- Method of Disposal

Original samples are stored in following areas of the laboratory.

1. Blue Air Refrigerator in Sample Storage Room: All water VOCs must be stored in the refrigerator.
2. Walk In Refrigerator in Sample Storage Room: All waters for all analyses except VOCs must be stored in this refrigerator.
4. Soil Walkin Refrigerator for all soils.

All soils are treated as quarantined.

All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)
2. Highly contaminated waste or product type samples which could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Testing Coordinator for other options.

Any person removing samples from the storage areas listed above, must sign them out on a laboratory custody sheet (attached). The individual performing the processing becomes responsible for the samples at this point. The samples are

maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

Sample extracts and digestates are stored in the following areas:

1. All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.
2. Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.
3. Extracts from medium level VOC analyses are also stored in the Soil Walk – in or VOC sample freezer in the VOC Lab.
4. All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure. Samples will be held for a minimum of 30 days after the final report unless specified otherwise. Sample extracts and digestates are held for a minimum of 60 days after the final report unless project specific requirements state otherwise. See SOP No. 405 entitled Laboratory Waste Disposal SOP for guidance on disposal of samples.

The following personnel as of September 08, 2008 have access to all sample storage areas:

Chandra Arthur	Herbie Johnson
Ashley Bester	Dahae Kim
Roger Burr	Dustin Lynch
Tanisha Custer	Marcia McGinnity
Rick Davis	Badeen Mekael
Barbara Dawson	AntonioMontiero
Betty DeVille	Ashley Morris
Amanda Fei	Gino Moore
Kendra Gentry	E. J. Overby
Jason Goodman	Brenton Powers
Sonya Gordon	Brian Richard
Gwen Hallquist	Franklin Rivers
Andrew Holder	William Schwab
Jade Holliman	Christy Thompson
John Hughes	Renee Vogel
Karu Huka	Randy Ward

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

For additional information see SOP No. 404 entitled Laboratory Sample Receiving, Log-In and Storage.



# CERTIFICATE OF ACCREDITATION

**ANSI-ASQ National Accreditation Board/AClass**  
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

**APPL, Inc.**  
**908 N. Temperance Avenue**  
**Clovis, CA 93611**

has been assessed by AClass  
and meets the requirements of

**DoD-ELAP**

while demonstrating technical competence in the field(s) of

**TESTING**

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1410

Certificate Number

AClass Approval

Certificate Valid: 10/23/2009-10/23/2011  
Version No. 001





# ANSI-ASQ National Accreditation Board

## SCOPE OF DoD-ELAP ACCREDITATION

### APPL, Inc.

908 N. Temperance Avenue, Clovis, CA 93611  
 Diane Anderson Phone: 559-275-2175

### TESTING

Valid to: October 23, 2011

Certificate Number: ADE-1410

#### I. Environmental

MATRIX	SPECIFIC TEST or GROUP of ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water / Wastewater	Acid Digestion for Metals Analysis	3010A	
Solid / Solid Waste	Acid digestion for Metals Analysis	3050B	
Water / Wastewater	Mercury Digestion and Analysis	245.1 / 7470A	AAS
Solid / Solid Waste	Mercury Digestion and Analysis	7471B	AAS
Water / Wastewater	Microwave assisted Acid Digestion for Metals Analysis	3015	Microwave
Solid / Solid Waste	Microwave assisted Acid Digestion for Metals Analysis	3051A	Microwave
Water / Wastewater	Purge and Trap for Aqueous Samples	5030B / 5030C	
Solid / Solid Waste	Closed-system purge and trap extraction for VOA analysis	5035 / 5035A	
Water / Wastewater	Separatory Funnel Extraction	3510C	
Solid / Solid Waste	Ultrasonic Extraction	3550B	Ultrasonic waterbath
Solid / Solid Waste	Soxhlet Extraction	3540C	Soxhlet Extractors

<b>MATRIX</b>	<b>SPECIFIC TEST or GROUP of ANALYTES</b>	<b>SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)</b>	<b>* KEY EQUIPMENT OR TECHNOLOGY USED</b>
Water / Wastewater	Liquid-Liquid Extraction	3520C	Liquid-Liquid Extractor
Water / Wastewater / Solid / Solid Waste	Silica gel cleanup	3630C	
Solid / Solid Waste	Incremental sampling	8330B, Appendix A	Puck mill grinder
Water / Wastewater / Solid / Solid Waste	Sulfur cleanup	3660B	
Water / Wastewater / Solid / Solid Waste	Sulfuric acid – permanganate cleanup	3665A	
Water / Wastewater / Solid / Solid Waste	Gel permeation cleanup	3640A	
Solid / Solid Waste	TCLP extraction	1311	Rotary Tumbler
Solid / Solid Waste	SPLP extraction	1312	Rotary Tumbler
Solid / Solid Waste	Waste Extraction Test (WET)	CCR Chapter 11, Article 5, Appendix II	Rotary Tumbler
Water / Wastewater	Total Dissolved Solids	160.1 / 2540C	Gravimetric
Water / Wastewater	Total Suspended Solids	160.2 / 2540D	Gravimetric
Water / Wastewater	Anion analysis	300.0 / 9056 / 9056A	Dionex Ion Chromatography
Solid / Solid Waste	Anion analysis	9056 / 9056A	Dionex Ion Chromatography
Water / Wastewater / Solid / Solid Waste	Perchlorate analysis	314.0	Dionex Ion Chromatography
Water / Wastewater / Solid / Solid Waste	Ammonia	350.1	Lachat Flow Injection Analysis
Water / Wastewater / Solid / Solid Waste	TKN	351.2	Lachat Flow Injection Analysis
Water / Wastewater / Solid / Solid Waste	Nitrate / Nitrite	353.2	Lachat Flow Injection Analysis
Water / Wastewater / Solid / Solid Waste	Sulfide	376.1	Titrimetric
Water	Fluoride	9214	Ion Selective Electrode

<b>MATRIX</b>	<b>SPECIFIC TEST or GROUP of ANALYTES</b>	<b>SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)</b>	<b>* KEY EQUIPMENT OR TECHNOLOGY USED</b>
Drinking Water / Water / Wastewater / Solid / Solid Waste	PCB Congeners	1668	High Resolution GC/MS
Water / Wastewater / Solid / Solid Waste	Perchlorate	6850	HPLC/Electrospray Ionization/MS
Water / Wastewater	Oil & Grease	1664A	Gravimetric
Water / Wastewater	Oil & Grease	5520B	Gravimetric
Water / Wastewater	TRPH	5520BF	Gravimetric
Water / Wastewater / Solid / Solid Waste	Total Metals	6010B / 6010C	ICP
Water / Wastewater / Solid / Solid Waste	Total Metals	6020 / 6020A	ICP/MS
Water / Wastewater / Solid / Solid Waste	Hexavalent Chromium	7196A	UV/Vis
Solid / Solid Waste	Alkaline digestion of Hexavalent Chromium	3060A	
Water / Wastewater	Hexavalent Chromium	218.6 / 7199	Dionex Ion Chromatography
Water / Wastewater / Solid / Solid Waste	Total Cyanide Distillation	9010B / 9010C	Midi-Distillation unit
Water / Wastewater / Solid / Solid Waste	Total Cyanide Analysis	9014	UV/Vis
Water / Wastewater	Corrosivity - pH	9040B	Ion Selective Electrode
Solid / Solid Waste	Corrosivity - pH	9045C / 9045D	Ion Selective Electrode
Water / Wastewater / Solid / Solid Waste	Chlorinated & Brominated Hydrocarbons	8011	GC/ECD
Water / Wastewater / Solid / Solid Waste	DRO/GRO	8015B/C/D	GC/FID
Water / Wastewater / Solid / Solid Waste	BTEX	8021B	GC/PID
Water / Solid	OP Pesticides	614 / 8141A / 8141B	GC/ECD
Water / Waste Water	OP Pesticides	614	GC/ECD

<b>MATRIX</b>	<b>SPECIFIC TEST or GROUP of ANALYTES</b>	<b>SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)</b>	<b>* KEY EQUIPMENT OR TECHNOLOGY USED</b>
Water / Waste Water	OCL Pesticides	608	GC/ECD
Water / Wastewater / Solid / Solid Waste	OCL Pesticides	8081A / 8081B	GC/ECD
Water / Waste Water	PCB	608	GC/ECD
Water / Wastewater / Solid / Solid Waste	PCB	8082 / 8082A	GC/ECD
Water / Waste Water	Herbicides	615	GC/ECD
Water / Wastewater / Solid / Solid Waste	Herbicides	8151A	GC/ECD
Water / Wastewater / Solid / Solid Waste	VOA	8260B / 8260C	GC/MS
Water / Wastewater / Solid / Solid Waste	PAH	8270 SIM	GC/MS
Water / Waste Water	Semi-VOA	625	GC/MS
Water / Wastewater / Solid / Solid Waste	Semi-VOA	8270C / 8270D	GC/MS
Water / Wastewater / Solid / Solid Waste	Dioxins	8290	HRGC/HRMS
Water / Wastewater / Solid / Solid Waste	Nitroaromatics & Nitramines & Nitroguanidine	8330A / 8330B / 8321A&B	HPLC
Water / Wastewater / Solid / Solid Waste	Carbamates	8321A / 8321B	HPLC
Solid / Solid Waste	Ignitability	1030	

**Notes:**

1. \* = As Applicable
2. This scope is part of and must be included with the Certificate of Accreditation No. ADE- 1410



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Vice President

DoD ELAP -- PT Performance Summary Review -- WP ALL

Lab Name :		APPL, Inc.				
City/State :		Clovis, CA				
PT Provider Used :		ERA, Absolute, RTC, APG				
PartName	PartNumber	NELACCCode	AnalyteName	EPAmethod#	PT results - Pass/Acceptable	Results
pH	4060	1900	pH	EPA 150.2	Pass	
WP pH @ 25°C	55061	1900	pH	EPA 150.2	Pass	
Solids (Total Solids, TSS, & TDS)	55085	1955	Total Dissolved Solids (TDS)	EPA 160.1	Pass	
WP Minerals #1	55144	1955	Total Dissolved Solids @ 180°C	EPA 160.1	Pass	
Solids	4030	1705	Total Dissolved Solids at 180C	EPA 160.1	Pass	
Solids (Total Solids, TSS, & TDS)	55085	1960	Non-Filterable Residue (TSS)	EPA 160.2	Pass	
Solids	4030	1960	Total Suspended Solids	EPA 160.2	Pass	
Oil & Grease	4120	1860	Oil & Grease	EPA 1664A	Pass	
Oil & Grease - n-Hexadecane & Stearic acid	55084	1860	Oil & Grease	EPA 1664A	Pass	
PCB Congeners in Water	PEO-403	9070	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9025	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9040	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8980	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8955	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9085	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9050	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 156)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9045	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8985	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9055	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9005	2,3,4,4',5'-Pentachlorobiphenyl (PCB 114)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8995	2,3',4,4',5'-Pentachlorobiphenyl (PCB 118)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9000	2,3',4,4',5'-Pentachlorobiphenyl (PCB 123)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8936	2,4,4'-Trichlorobiphenyl (PCB 28)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9060	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9015	3,3',4,4',5'-Pentachlorobiphenyl (PCB 126)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8965	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8970	3,4,4',5'-Tetrachlorobiphenyl (PCB 81)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9025	PCB (129)+(138)+(163)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9040	PCB (153)+(168)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9046	PCB (156)+(157)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	9070	PCB (180)+(193)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8936	PCB (20)+(28)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8980	PCB (90)+(101)+(113)	EPA 1668	Pass	
PCB Congeners in Water	PEO-403	8870	PCBs, total	EPA 1668	Pass	
Bromide	4850	1540	Bromide	EPA 300.0	Pass	
CWA Anions	55131	1540	Bromide (Br)	EPA 300.0	Pass	
Minerals	4050	1575	Chloride	EPA 300.0	Pass	
WP Minerals #1	55144	1575	Chloride	EPA 300.0	Pass	
Fluoride	4420	1730	Fluoride	EPA 300.0	Pass	
WP Minerals #2	55145	1730	Fluoride	EPA 300.0	Pass	
WP & DMRQA Nutrients	55035	1810	Nitrate as N	EPA 300.0	Pass	
WP Nitrate & Nitrite	55130	1810	Nitrate as N	EPA 300.0	Pass	
Nutrients	4020	1810	Nitrate Nitrogen as N	EPA 300.0	Pass	
Nitrate-Nitrite as N	4770	1820	Nitrate-Nitrite as N	EPA 300.0	Pass	
WP Nitrate & Nitrite	55130	1820	Nitrite + Nitrate as N	EPA 300.0	Pass	
Nitrite as N	4780	1840	Nitrite as N	EPA 300.0	Pass	
WP Nitrate & Nitrite	55130	1840	Nitrite as N	EPA 300.0	Pass	
Nutrients	4020	1870	Orthophosphate as P	EPA 300.0	Pass	
WP & DMRQA Nutrients	55035	1870	Orthophosphate as P	EPA 300.0	Pass	
Minerals	4050	2000	Sulfate	EPA 300.0	Pass	
WP Minerals #2	55145	2000	Sulfate	EPA 300.0	Pass	
Miscellaneous Analytes	PEI-051	1540	Bromide	EPA 300.0	Pass	
Minerals	PEI-051	1575	Chloride	EPA 300.0	Pass	
Minerals	PEI-051	1730	Fluoride	EPA 300.0	Pass	
Nutrients	PEI-051	1805	Nitrate as N	EPA 300.0	Pass	
Nutrients	PEI-051	1820	Nitrate+nitrite as N	EPA 300.0	Pass	
Nutrients	PEI-051	1840	Nitrite as N	EPA 300.0	Pass	
Nutrients	PEI-051	1870	Orthophosphate as P	EPA 300.0	Pass	
Minerals	PEI-051	2000	Sulfate	EPA 300.0	Pass	
WP Perchlorate	55116	1895	Perchlorate	EPA 314.0	Pass	
Fluoride	4420	1730	Fluoride	EPA 340.2	Pass	
WP Minerals #2	55145	1730	Fluoride	EPA 340.2	Pass	
WP & DMRQA Nutrients	55035	1515	Ammonia as N	EPA 350.1	Pass	
Nutrients	4020	1515	Ammonia Nitrogen as N	EPA 350.1	Pass	
Nutrients	4020	1795	Total Kjeldahl Nitrogen	EPA 351.2	Pass	
WP & DMRQA Nutrients #2	55064	1795	Total Kjeldahl Nitrogen	EPA 351.2	Pass	
WP & DMRQA Nutrients	55035	1810	Nitrate as N	EPA 353.2	Pass	
WP Nitrate & Nitrite	55130	1810	Nitrate as N	EPA 353.2	Pass	
Nutrients	4020	1810	Nitrate Nitrogen as N	EPA 353.2	Pass	
Nitrate-Nitrite as N	4770	1820	Nitrate-Nitrite as N	EPA 353.2	Pass	
WP Nitrate & Nitrite	55130	1820	Nitrite + Nitrate as N	EPA 353.2	Pass	
Nitrite as N	4780	1840	Nitrite as N	EPA 353.2	Pass	
WP Nitrate & Nitrite	55130	1840	Nitrite as N	EPA 353.2	Pass	
Sulfide	4900	2005	Sulfide	EPA 376.1	Pass	
Sulphide	55042	2005	Sulphide	EPA 376.1	Pass	
MBAS	4430	2025	MBAS	EPA 425.1	Pass	
Trace Metals	4070	1000	Aluminum	EPA 6010B	Pass	
WP & DMRQA Trace Elements	55024	1000	Aluminum	EPA 6010B	Pass	
WP & DMRQA Trace Elements	55024	1000	Aluminum	EPA 6010B	Pass	
Trace Metals 1	PEI-034-1	1000	Aluminum, Al	EPA 6010B	Pass	
Trace Metals	4070	1005	Antimony	EPA 6010B	Pass	
WP Trace Elements	55025	1005	Antimony	EPA 6010B	Pass	
Trace Metals 2	PEI-034-2	1005	Antimony, Sb	EPA 6010B	Pass	

Trace Metals	4070	1010	Arsenic	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1010	Arsenic	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1010	Arsenic	EPA 6010B	Pass		
Trace Metals 1	PEI-034-1	1010	Arsenic, As	EPA 6010B	Pass		
WP Trace Elements	55025	1015	Barium	EPA 6010B	Pass		
Trace Metals	586	1015	Barium	EPA 6010B	Pass		
Trace Metals	4070	1020	Beryllium	EPA 6010B	Pass		
WP Trace Elements	55025	1020	Beryllium	EPA 6010B	Pass		
Trace Metals 1	PEI-034-1	1020	Beryllium, Be	EPA 6010B	Pass		
Trace Metals	4070	1025	Boron	EPA 6010B	Pass		
WP Trace Elements	55025	1025	Boron	EPA 6010B	Pass		
WP Trace Elements	55025	1025	Boron	EPA 6010B	Pass		
Trace Metals 2	PEI-034-2	1025	Boron, B	EPA 6010B	Pass		
Trace Metals	4070	1030	Cadmium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1030	Cadmium	EPA 6010B	Pass		
WP Minerals #1	55144	1035	Calcium	EPA 6010B	Pass		
WP Minerals #1	55144	1550	Calcium Hardness (CaCO3)	EPA 6010B	Pass		
Trace Metals	4070	1040	Chromium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1040	Chromium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1050	Cobalt	EPA 6010B	Pass		
Trace Metals	586	1050	Cobalt	EPA 6010B	Pass		
Trace Metals	4070	1055	Copper	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1055	Copper	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1055	Copper	EPA 6010B	Pass		
Trace Metals	4070	1070	Iron	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1070	Iron	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1070	Iron	EPA 6010B	Pass		
Trace Metals	4070	1075	Lead	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1075	Lead	EPA 6010B	Pass		
WP Minerals #1	55144	1085	Magnesium	EPA 6010B	Pass		
Trace Metals	4070	1090	Manganese	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1090	Manganese	EPA 6010B	Pass		
Trace Metals 1	PEI-034-1	1090	Manganese, Mn	EPA 6010B	Pass		
Trace Metals	4070	1100	Molybdenum	EPA 6010B	Pass		
WP Trace Elements	55025	1100	Molybdenum	EPA 6010B	Pass		
WP Trace Elements	55025	1100	Molybdenum	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1105	Nickel	EPA 6010B	Pass		
Trace Metals	586	1105	Nickel	EPA 6010B	Pass		
WP Minerals #2	55145	1125	Potassium	EPA 6010B	Pass		
Trace Metals	4070	1140	Selenium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1140	Selenium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1140	Selenium	EPA 6010B	Pass		
Trace Metals 1	PEI-034-1	1140	Selenium, Se	EPA 6010B	Pass		
Trace Metals	4070	1150	Silver	EPA 6010B	Pass		
WP Trace Elements	55025	1150	Silver	EPA 6010B	Pass		
WP Trace Elements	55025	1150	Silver	EPA 6010B	Pass		
Trace Metals 2	PEI-034-2	1150	Silver, Ag	EPA 6010B	Pass		
WP Minerals #2	55145	1155	Sodium	EPA 6010B	Pass		
Trace Metals	4070	1160	Strontium	EPA 6010B	Pass		
WP Trace Elements	55025	1160	Strontium	EPA 6010B	Pass		
WP Trace Elements	55025	1160	Strontium	EPA 6010B	Pass		
Trace Metals 2	PEI-034-2	1160	Strontium, Sr	EPA 6010B	Pass		
Trace Metals	4070	1165	Thallium	EPA 6010B	Pass		
WP Trace Elements	55025	1165	Thallium	EPA 6010B	Pass		
WP Trace Elements	55025	1165	Thallium	EPA 6010B	Pass		
Trace Metals	4070	1175	Tin	EPA 6010B	Pass		
WP Tin	55095	1175	Tin	EPA 6010B	Pass		
Barium & Tin	PEI-034-5	1175	Tin, Sn	EPA 6010B	Pass		
Trace Metals	4070	1180	Titanium	EPA 6010B	Pass		
WP Trace Elements	55025	1180	Titanium	EPA 6010B	Pass		
WP Trace Elements	55025	1180	Titanium	EPA 6010B	Pass		
Trace Metals 2	PEI-034-2	1180	Titanium, Ti	EPA 6010B	Pass		
WP Minerals #1	55144	1755	Total Hardness (CaCO3)	EPA 6010B	Pass		
Trace Metals	4070	1185	Vanadium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1185	Vanadium	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1185	Vanadium	EPA 6010B	Pass		
Trace Metals	4070	1190	Zinc	EPA 6010B	Pass		
WP & DMRQA Trace Elements	55024	1190	Zinc	EPA 6010B	Pass		
Trace Metals	4070	1000	Aluminum	EPA 6020	Pass		
Trace Metals	4070	1005	Antimony	EPA 6020	Pass		
WP Trace Elements	55025	1005	Antimony	EPA 6020	Pass		
Trace Metals	4070	1010	Arsenic	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1010	Arsenic	EPA 6020	Pass		
Trace Metals	4070	1015	Barium	EPA 6020	Pass		
Trace Metals	4070	1020	Beryllium	EPA 6020	Pass		
WP Trace Elements	55025	1020	Beryllium	EPA 6020	Pass		
Trace Metals	4070	1025	Boron	EPA 6020	Pass		
Trace Metals	586	1025	Boron	EPA 6020	Pass		
Trace Metals	PEI-034	1025	Boron, B	EPA 6020	Pass		
Trace Metals	4070	1030	Cadmium	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1030	Cadmium	EPA 6020	Pass		
Trace Metals	4070	1040	Chromium	EPA 6020	Pass		
Trace Metals	4070	1050	Cobalt	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1050	Cobalt	EPA 6020	Pass		
Trace Metals	4070	1055	Copper	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1055	Copper	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1070	Iron	EPA 6020	Pass		
Trace Metals	586	1070	Iron	EPA 6020	Pass		
Trace Metals	4070	1075	Lead	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1075	Lead	EPA 6020	Pass		
Trace Metals	4070	1090	Manganese	EPA 6020	Pass		

WP & DMRQA Trace Elements	55024	1090	Manganese	EPA 6020	Pass		
Trace Metals	4070	1100	Molybdenum	EPA 6020	Pass		
WP Trace Elements	55025	1100	Molybdenum	EPA 6020	Pass		
Trace Metals	4070	1105	Nickel	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1105	Nickel	EPA 6020	Pass		
Trace Metals	4070	1140	Selenium	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1140	Selenium	EPA 6020	Pass		
Trace Metals	4070	1150	Silver	EPA 6020	Pass		
Trace Metals	4070	1160	Strontium	EPA 6020	Pass		
WP Trace Elements	55025	1160	Strontium	EPA 6020	Pass		
Trace Metals	4070	1165	Thallium	EPA 6020	Pass		
WP Trace Elements	55025	1165	Thallium	EPA 6020	Pass		
Trace Metals	4070	1175	Tin	EPA 6020	Pass		
WP Trace Elements	55025	1180	Titanium	EPA 6020	Pass		
Trace Metals	4070	1185	Vanadium	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1185	Vanadium	EPA 6020	Pass		
Trace Metals	4070	1190	Zinc	EPA 6020	Pass		
WP & DMRQA Trace Elements	55024	1190	Zinc	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1000	Aluminum, Al	EPA 6020	Pass		
Trace Metals 2	PEI-034-2	1005	Antimony, Sb	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1010	Arsenic, As	EPA 6020	Pass		
Barium & Tin	PEI-034-5	1015	Barium, Ba	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1020	Beryllium, Be	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1030	Cadmium, Cd	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1040	Chromium, Cr (total)	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1050	Cobalt, Co	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1055	Copper, Cu	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1070	Iron, Fe	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1075	Lead, Pb	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1090	Manganese, Mn	EPA 6020	Pass		
Trace Metals 2	PEI-034-2	1100	Molybdenum, Mo	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1105	Nickel, Ni	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1140	Selenium, Se	EPA 6020	Pass		
Trace Metals 2	PEI-034-2	1150	Silver, Ag	EPA 6020	Pass		
Barium & Tin	PEI-034-5	1175	Tin, Sn	EPA 6020	Pass		
Trace Metals 2	PEI-034-2	1180	Titanium, Ti	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1185	Vanadium, V	EPA 6020	Pass		
Trace Metals 1	PEI-034-1	1190	Zinc, Zn	EPA 6020	Pass		
Pesticides (WP)	4460	7355	4,4'-DDD	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7355	4,4'-DDD	EPA 608	Pass		
Pesticides (WP)	4460	7360	4,4'-DDE	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7360	4,4'-DDE	EPA 608	Pass		
Pesticides (WP)	4460	7365	4,4'-DDT	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7365	4,4'-DDT	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7810	4,4'-Methoxychlor	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7110	a-BHC	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7240	a-Chlordane	EPA 608	Pass		
Pesticides (WP)	4460	7025	Aldrin	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7025	Aldrin	EPA 608	Pass		
Pesticides (NELAC)	4460	7110	alpha-BHC	EPA 608	Pass		
Pesticides (NELAC)	4460	7240	alpha-Chlordane	EPA 608	Pass		
WP PCBs in Water	38091	8880	Aroclor 1016	EPA 608	Pass		
WP PCBs in Water	38094	8880	Aroclor 1016	EPA 608	Pass		
WP PCBs in Water	832S	8880	Aroclor 1016	EPA 608	Pass		
PCBs in Water	4130	8880	Aroclor 1016	EPA 608	Pass		
PCBs in Oil	4140	8880	Aroclor 1016 in Oil	EPA 608	Pass		
PCBs in Water	4130	8880	Aroclor 1016 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8880	Aroclor 1016 Sample 2	EPA 608	Pass		
PCBs in Water	832S	8885	Aroclor 1221	EPA 608	Pass		
WP PCBs in Water	38091	8885	Aroclor 1221	EPA 608	Pass		
WP PCBs in Water	38094	8885	Aroclor 1221	EPA 608	Pass		
PCBs in Water	4130	8885	Aroclor 1221	EPA 608	Pass		
PCBs in Oil	4140	8885	Aroclor 1221 in Oil	EPA 608	Pass		
WP PCBs in Water	38091	8890	Aroclor 1232	EPA 608	Pass		
WP PCBs in Water	38094	8890	Aroclor 1232	EPA 608	Pass		
WP PCBs in Water	832S	8890	Aroclor 1232	EPA 608	Pass		
PCBs in Water	4130	8890	Aroclor 1232	EPA 608	Pass		
PCBs in Oil	4140	8890	Aroclor 1232 in Oil	EPA 608	Pass		
PCBs in Water	4130	8890	Aroclor 1232 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8890	Aroclor 1232 Sample 2	EPA 608	Pass		
WP PCBs in Water	832S	8895	Aroclor 1242	EPA 608	Pass		
WP PCBs in Water	38091	8895	Aroclor 1242	EPA 608	Pass		
WP PCBs in Water	38094	8895	Aroclor 1242	EPA 608	Pass		
PCBs in Water	4130	8895	Aroclor 1242	EPA 608	Pass		
PCBs in Oil	4140	8895	Aroclor 1242 in Oil	EPA 608	Pass		
PCBs in Water	4130	8895	Aroclor 1242 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8895	Aroclor 1242 Sample 2	EPA 608	Pass		
WP PCBs in Water	38091	8900	Aroclor 1248	EPA 608	Pass		
WP PCBs in Water	38094	8900	Aroclor 1248	EPA 608	Pass		
PCBs in Oil	4140	8900	Aroclor 1248 in Oil	EPA 608	Pass		
PCBs in Water	4130	8900	Aroclor 1248 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8900	Aroclor 1248 Sample 2	EPA 608	Pass		
PCBs in Water	832S	8905	Aroclor 1254	EPA 608	Pass		
WP PCBs in Water	38091	8905	Aroclor 1254	EPA 608	Pass		
WP PCBs in Water	38094	8905	Aroclor 1254	EPA 608	Pass		
PCBs in Water	4130	8905	Aroclor 1254	EPA 608	Pass		
PCBs in Oil	4140	8905	Aroclor 1254 in Oil	EPA 608	Pass		
PCBs in Water	4130	8905	Aroclor 1254 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8905	Aroclor 1254 Sample 2	EPA 608	Pass		
WP PCBs in Water	38091	8910	Aroclor 1260	EPA 608	Pass		
WP PCBs in Water	38094	8910	Aroclor 1260	EPA 608	Pass		

WP PCBs in Water	832S	8910	Aroclor 1260	EPA 608	Pass		
PCBs in Water	4130	8910	Aroclor 1260	EPA 608	Pass		
PCBs in Oil	4140	8910	Aroclor 1260 in Oil	EPA 608	Pass		
PCBs in Water	4130	8910	Aroclor 1260 Sample 1	EPA 608	Pass		
PCBs in Water	4130	8910	Aroclor 1260 Sample 2	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7115	b-BHC	EPA 608	Pass		
Pesticides (NELAC)	4460	7115	beta-BHC	EPA 608	Pass		
WP Pesticide Amp 2	38046	7250	Chlordane (total)	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7105	d-BHC	EPA 608	Pass		
Pesticides (NELAC)	4460	7105	delta-BHC	EPA 608	Pass		
Pesticides (WP)	4460	7470	Dieldrin	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7470	Dieldrin	EPA 608	Pass		
Pesticides (NELAC)	4460	7510	Endosulfan I	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7510	Endosulfan I	EPA 608	Pass		
Pesticides (NELAC)	4460	7515	Endosulfan II	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7515	Endosulfan II	EPA 608	Pass		
Pesticides (NELAC)	4460	7520	Endosulfan sulfate	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7520	Endosulfan sulfate	EPA 608	Pass		
Pesticides (NELAC)	4460	7540	Endrin	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7540	Endrin	EPA 608	Pass		
Pesticides (NELAC)	4460	7530	Endrin aldehyde	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7530	Endrin aldehyde	EPA 608	Pass		
Pesticides (NELAC)	4460	7535	Endrin Ketone	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7535	Endrin ketone	EPA 608	Pass		
Pesticides (NELAC)	4460	7120	gamma-BHC (Lindane)	EPA 608	Pass		
Pesticides (NELAC)	4460	7245	gamms-Chlordane	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7120	g-BHC (Lindane)	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7245	g-Chlordane	EPA 608	Pass		
Pesticides (WP)	4460	7685	Heptachlor	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7685	Heptachlor	EPA 608	Pass		
Pesticides (WP)	4460	7690	Heptachlor epoxide	EPA 608	Pass		
WP Organochlorine Pesticides	38122	7690	Heptachlor epoxide	EPA 608	Pass		
Pesticides (NELAC)	4460	7810	Methoxychlor	EPA 608	Pass		
WP PCBs in Transformer Oil	38092	8880	PCB in Oil 1016 or 1242	EPA 608	Pass		
WP PCBs in Water	38094	8880	PCB in Oil 1016 or 1242	EPA 608	Pass		
WP PCBs in Transformer Oil	38092	8905	PCB in Oil 1254	EPA 608	Pass		
WP PCBs in Water	38094	8905	PCB in Oil 1254	EPA 608	Pass		
WP PCBs in Transformer Oil	38092	8910	PCB in Oil 1260	EPA 608	Pass		
WP PCBs in Water	38094	8910	PCB in Oil 1260	EPA 608	Pass		
Total Chlordane	4160	7250	Total Chlordane	EPA 608	Pass		
Toxaphene	4270	8250	Toxaphene	EPA 608	Pass		
WP Acrolein & Acrylonitrile	38123	8250	Toxaphene	EPA 608	Pass		
Herbicides	4440	8655	2,4,5-T	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	8655	2,4,5-T	EPA 615	Pass		
Herbicides	4440	8650	2,4,5-TP (Silvex)	EPA 615	Pass		
Herbicides	4440	8545	2,4-D	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8545	2,4-D (2,4-Dichlorophenoxyacetic acid)	EPA 615	Pass		
Herbicides	4440	8560	2,4-DB	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	8560	2,4-DB	EPA 615	Pass		
Herbicides	4440	8600	3,5-Dichlorobenzoic acid	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	8600	3,5-Dichlorobenzoic acid	EPA 615	Pass		
Herbicides	4440	6500	4-Nitrophenol	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	6500	4-Nitrophenol	EPA 615	Pass		
Herbicides	4440	8505	Acifluorfen	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8505	Acifluorfen	EPA 615	Pass		
Herbicides	4440	8530	Bentazon	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	8530	Bentazon	EPA 615	Pass		
Herbicides	4440	8540	Chloramben	EPA 615	Pass		
Herbicides	4440	8550	Dacthal diacid (DCPA)	EPA 615	Pass		
Herbicides	4440	8555	Dalapon	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8555	Dalapon	EPA 615	Pass		
Herbicides	4440	8595	Dicamba	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8595	Dicamba	EPA 615	Pass		
Herbicides	4440	8605	Dichloroprop	EPA 615	Pass		
WP Herbicide Acid Mix #2	38136	8605	Dichloroprop	EPA 615	Pass		
Herbicides	4440	8620	Dinoseb	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8620	Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	EPA 615	Pass		
Herbicides	4440	7775	MCPA	EPA 615	Pass		
Herbicides	4440	7780	MCPP	EPA 615	Pass		
Herbicides	4440	6605	Pentachlorophenol	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	6605	Pentachlorophenol	EPA 615	Pass		
Herbicides	4440	8645	Picloram	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8645	Picloram	EPA 615	Pass		
WP Acrolein & Acrylonitrile	38123	8650	Silvex (2,4,5-TP)	EPA 615	Pass		
Volatiles	4170	5105	1,1,1,2-Tetrachloroethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5105	1,1,1,2-Tetrachloroethane	EPA 624	Pass		
Volatiles	4170	5160	1,1,1-Trichloroethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5160	1,1,1-Trichloroethane	EPA 624	Pass		
Volatiles	4170	5110	1,1,2,2-Tetrachloroethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5110	1,1,2,2-Tetrachloroethane	EPA 624	Pass		
Volatiles	4170	5165	1,1,2-Trichloroethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5165	1,1,2-Trichloroethane	EPA 624	Pass		
WP Oxygenates	38157	5185	1,1,2-Trichlorotrifluoroethane	EPA 624	Pass		
Volatiles	4170	4630	1,1-Dichloroethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4630	1,1-Dichloroethane	EPA 624	Pass		
Volatiles	4170	4640	1,1-Dichloroethene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4640	1,1-Dichloroethene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4670	1,1-Dichloropropene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5150	1,2,3-Trichlorobenzene	EPA 624	Pass		
Volatiles	4170	5180	1,2,3-Trichloropropane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5180	1,2,3-Trichloropropane	EPA 624	Pass		

Volatiles in Non-Potable Water	38083	5210	1,2,4-Trimethylbenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4570	1,2-Dibromo-3-chloropropane	EPA 624	Pass
Volatiles	4170	4585	1,2-Dibromoethane (EDB)	EPA 624	Pass
Volatiles Aromatics	4450	4610	1,2-Dichlorobenzene	EPA 624	Pass
Volatiles	4170	4610	1,2-Dichlorobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4610	1,2-Dichlorobenzene	EPA 624	Pass
Volatiles	4170	4635	1,2-Dichloroethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4635	1,2-Dichloroethane	EPA 624	Pass
Volatiles	4170	4655	1,2-Dichloropropane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4655	1,2-Dichloropropane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	5215	1,3,5-Trimethylbenzene	EPA 624	Pass
Volatiles Aromatics	4450	4615	1,3-Dichlorobenzene	EPA 624	Pass
Volatiles	4170	4615	1,3-Dichlorobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4615	1,3-Dichlorobenzene	EPA 624	Pass
Volatiles Aromatics	4450	4620	1,4-Dichlorobenzene	EPA 624	Pass
Volatiles	4170	4620	1,4-Dichlorobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4620	1,4-Dichlorobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4665	2,2-Dichloropropane	EPA 624	Pass
Volatiles	4170	4410	2-Butanone (Methyl ethyl ketone)	EPA 624	Pass
WP Ketones	38134	4410	2-Butanone	EPA 624	Pass
Volatiles	4170	4500	2-Chloroethyl vinyl ether	EPA 624	Pass
WP 2-Chloroethyl vinyl ether	38128	4500	2-Chloroethyl vinyl ether	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4535	2-Chlorotoluene	EPA 624	Pass
Volatiles	4170	4860	2-Hexanone	EPA 624	Pass
WP Ketones	38134	4860	2-Hexanone	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4540	4-Chlorotoluene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4995	4-Methyl-2-pentanone	EPA 624	Pass
WP Ketones	38134	4995	4-Methyl-2-pentanone	EPA 624	Pass
Volatiles	4170	4995	4-Methyl-2-pentanone (MIBK)	EPA 624	Pass
Volatiles	4170	4315	Acetone	EPA 624	Pass
WP Ketones	38134	4315	Acetone	EPA 624	Pass
Volatiles	4170	4325	Acrolein	EPA 624	Pass
WP Acrolein & Acrylonitrile	38123	4325	Acrolein	EPA 624	Pass
Volatiles	4170	4340	Acrylonitrile	EPA 624	Pass
WP Acrolein & Acrylonitrile	38123	4340	Acrylonitrile	EPA 624	Pass
CWA BTEX & MTBE	38166	4375	Benzene	EPA 624	Pass
Volatiles Aromatics	4450	4375	Benzene	EPA 624	Pass
Volatiles	4170	4375	Benzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4375	Benzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4385	Bromobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4390	Bromochloromethane	EPA 624	Pass
Volatiles	4170	4395	Bromodichloromethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4395	Bromodichloromethane	EPA 624	Pass
Volatiles	4170	4400	Bromoform	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4400	Bromoform	EPA 624	Pass
Volatiles	4170	4950	Bromomethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4950	Bromomethane	EPA 624	Pass
Volatiles	4170	4450	Carbon disulfide	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4450	Carbon disulfide	EPA 624	Pass
Volatiles	4170	4455	Carbon tetrachloride	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4455	Carbon tetrachloride	EPA 624	Pass
Volatiles	4170	4475	Chlorobenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4475	Chlorobenzene	EPA 624	Pass
Volatiles	4170	4485	Chloroethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4485	Chloroethane	EPA 624	Pass
Volatiles	4170	4505	Chloroform	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4505	Chloroform	EPA 624	Pass
Volatiles	4170	4960	Chloromethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4960	Chloromethane	EPA 624	Pass
Volatiles	4170	4645	cis-1,2-Dichloroethene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4645	cis-1,2-Dichloroethene	EPA 624	Pass
Volatiles	4170	4680	cis-1,3-Dichloropropene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4680	cis-1,3-Dichloropropene	EPA 624	Pass
Volatiles	4170	4575	Dibromochloromethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4575	Dibromochloromethane	EPA 624	Pass
Volatiles	4170	4595	Dibromomethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4595	Dibromomethane	EPA 624	Pass
Volatiles	4170	4625	Dichlorodifluoromethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4625	Dichlorodifluoromethane	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4765	Ethyl benzene	EPA 624	Pass
CWA BTEX & MTBE	38166	4765	Ethylbenzene	EPA 624	Pass
Volatiles Aromatics	4450	4765	Ethylbenzene	EPA 624	Pass
Volatiles	4170	4765	Ethylbenzene	EPA 624	Pass
Volatiles	4170	4835	Hexachlorobutadiene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4835	Hexachlorobutadiene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4840	Hexachloroethane	EPA 624	Pass
WP Oxygenates	38157	9375	Isopropyl ether (DIPE)	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4900	Isopropylbenzene	EPA 624	Pass
CWA BTEX & MTBE	38166	5000	Methyl tert-butyl ether (MTBE)	EPA 624	Pass
Volatiles in Non-Potable Water	38083	5000	Methyl tert-butyl ether (MTBE)	EPA 624	Pass
WP Oxygenates	38157	5000	Methyl tert-butyl ether (MTBE)	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4975	Methylene chloride	EPA 624	Pass
Volatiles	4170	4975	Methylene chloride (Dichloromethane)	EPA 624	Pass
Volatiles	4170	5000	Methyl-t-butylether (MTBE)	EPA 624	Pass
Volatiles	4170	5005	Naphthalene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	5005	Naphthalene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4435	n-Butyl benzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	5090	n-Propylbenzene	EPA 624	Pass
WP Oxygenates	38157	5090	n-Propylbenzene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4910	p-Isopropyl toluene	EPA 624	Pass
Volatiles in Non-Potable Water	38083	4440	sec-Butyl benzene	EPA 624	Pass

Volatiles	4170	5100	Styrene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5100	Styrene	EPA 624	Pass		
WP Oxygenates	38157	4370	tert-Amyl methyl ether (TAME)	EPA 624	Pass		
WP Oxygenates	38157	4420	tert-Butyl alcohol (t-Butanol)	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4445	tert-Butyl benzene	EPA 624	Pass		
WP Oxygenates	38157	4770	tert-Butyl ethyl ether (ETBE)	EPA 624	Pass		
Volatiles	4170	5115	Tetrachloroethene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5115	Tetrachloroethene	EPA 624	Pass		
CWA BTEX & MTBE	38166	5140	Toluene	EPA 624	Pass		
Volatile Aromatics	4450	5140	Toluene	EPA 624	Pass		
Volatiles	4170	5140	Toluene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5140	Toluene	EPA 624	Pass		
CWA BTEX & MTBE	38166	5260	Total Xylenes	EPA 624	Pass		
Volatile Aromatics	4450	5260	Total Xylenes	EPA 624	Pass		
Volatiles	4170	5260	Total Xylenes	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5260	Total Xylenes	EPA 624	Pass		
Volatiles	4170	4700	trans-1,2-Dichloroethene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4700	trans-1,2-Dichloroethene	EPA 624	Pass		
Volatiles	4170	4685	trans-1,3-Dichloropropene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	4685	trans-1,3-Dichloropropene	EPA 624	Pass		
Volatiles	4170	5170	Trichloroethene	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5170	Trichloroethene	EPA 624	Pass		
Volatiles	4170	5175	Trichlorofluoromethane	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5175	Trichlorofluoromethane	EPA 624	Pass		
Volatiles	4170	5225	Vinyl acetate	EPA 624	Pass		
Volatiles	4170	5235	Vinyl Chloride	EPA 624	Pass		
Volatiles in Non-Potable Water	38083	5235	Vinyl chloride	EPA 624	Pass		
Base Neutral Extractables	4200	6715	1,2,4,5-Tetrachlorobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	5155	1,2,4-Trichlorobenzene	EPA 625	Pass		
WP Base/Neutrals	711	5155	1,2,4-Trichlorobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	4610	1,2-Dichlorobenzene	EPA 625	Pass		
WP Base/Neutrals	711	4610	1,2-Dichlorobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	4615	1,3-Dichlorobenzene	EPA 625	Pass		
WP Base/Neutrals	711	4615	1,3-Dichlorobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	4620	1,4-Dichlorobenzene	EPA 625	Pass		
WP Base/Neutrals	711	4620	1,4-Dichlorobenzene	EPA 625	Pass		
Acid Extractables	4190	6735	2,3,4,6-Tetrachlorophenol	EPA 625	Pass		
Acids	712	6735	2,3,4,6-Tetrachlorophenol	EPA 625	Pass		
Acid Extractables	4190	6835	2,4,5-Trichlorophenol	EPA 625	Pass		
Acids	712	6835	2,4,5-Trichlorophenol	EPA 625	Pass		
Acids	712	6840	2,4,6-Trichlorophenol	EPA 625	Pass		
Acid Extractables	4190	6840	2,4,6-Trichlorophenol	EPA 625	Pass		
Acid Extractables	4190	6000	2,4-Dichlorophenol	EPA 625	Pass		
Acids	712	6000	2,4-Dichlorophenol	EPA 625	Pass		
Acid Extractables	4190	6130	2,4-Dimethylphenol	EPA 625	Pass		
Acids	712	6130	2,4-Dimethylphenol	EPA 625	Pass		
Acid Extractables	4190	6175	2,4-Dinitrophenol	EPA 625	Pass		
Acids	712	6175	2,4-Dinitrophenol	EPA 625	Pass		
WP Base/Neutrals	711	6185	2,4-Dinitrotoluene	EPA 625	Pass		
Base Neutral Extractables	4200	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Pass		
Acid Extractables	4190	6005	2,6-Dichlorophenol	EPA 625	Pass		
Acids	712	6005	2,6-Dichlorophenol	EPA 625	Pass		
WP Base/Neutrals	711	6190	2,6-Dinitrotoluene	EPA 625	Pass		
Base Neutral Extractables	4200	6190	2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Pass		
Base Neutral Extractables	4200	5795	2-Chloronaphthalene	EPA 625	Pass		
WP Base/Neutrals	711	5795	2-Chloronaphthalene	EPA 625	Pass		
Acid Extractables	4190	5800	2-Chlorophenol	EPA 625	Pass		
Acids	712	5800	2-Chlorophenol	EPA 625	Pass		
Acid Extractables	4190	6360	2-Methyl-4,6-Dinitrophenol	EPA 625	Pass		
Base Neutral Extractables	4200	6385	2-Methylnaphthalene	EPA 625	Pass		
WP Base/Neutrals	711	6385	2-Methylnaphthalene	EPA 625	Pass		
Acid Extractables	4190	6400	2-Methylphenol	EPA 625	Pass		
Acids	712	6400	2-Methylphenol	EPA 625	Pass		
WP Base/Neutrals	711	6460	2-Nitroaniline	EPA 625	Pass		
Base Neutral Extractables	4200	6460	2-Nitroaniline	EPA 625	Pass		
Acid Extractables	4190	6490	2-Nitrophenol	EPA 625	Pass		
Acids	712	6490	2-Nitrophenol	EPA 625	Pass		
Acid Extractables	4190	6410	3 & 4-Methylphenol	EPA 625	Pass		
Base Neutral Extractables	4200	5945	3,3'-Dichlorobenzidine	EPA 625	Pass		
WP Base/Neutrals	711	5945	3,3'-Dichlorobenzidine	EPA 625	Pass		
Acid Extractables	4190	6405	3-Methylphenol	EPA 625	Pass		
Base Neutral Extractables	4200	6465	3-Nitroaniline	EPA 625	Pass		
WP Base/Neutrals	711	6465	3-Nitroaniline	EPA 625	Pass		
Acids	712	6360	4,6-Dinitro-2-methylphenol	EPA 625	Pass		
Base Neutral Extractables	4200	5660	4-Bromophenyl phenyl ether	EPA 625	Pass		
WP Base/Neutrals	711	5660	4-Bromophenyl-phenylether	EPA 625	Pass		
Acid Extractables	4190	5700	4-Chloro-3-methylphenol	EPA 625	Pass		
Acids	712	5700	4-Chloro-3-methylphenol	EPA 625	Pass		
Base Neutral Extractables	4200	5745	4-Chloroaniline	EPA 625	Pass		
WP Base/Neutrals	711	5745	4-Chloroaniline	EPA 625	Pass		
Base Neutral Extractables	4200	5825	4-Chlorophenyl-phenylether	EPA 625	Pass		
WP Base/Neutrals	711	5825	4-Chlorophenyl-phenylether	EPA 625	Pass		
Acids	712	6410	4-Methylphenol	EPA 625	Pass		
Base Neutral Extractables	4200	6470	4-Nitroaniline	EPA 625	Pass		
WP Base/Neutrals	711	6470	4-Nitroaniline	EPA 625	Pass		
Acid Extractables	4190	6500	4-Nitrophenol	EPA 625	Pass		
Acids	712	6500	4-Nitrophenol	EPA 625	Pass		
Base Neutral Extractables	4200	5500	Acenaphthene	EPA 625	Pass		
PAH-GC & GCMS	4880	5500	Acenaphthene	EPA 625	Pass		
WP Base/Neutrals	711	5500	Acenaphthene	EPA 625	Pass		
Base Neutral Extractables	4200	5505	Acenaphthylene	EPA 625	Pass		

PAH-GC & GCMS	4880	5505	Acenaphthylene	EPA 625	Pass		
WP Base/Neutrals	711	5505	Acenaphthylene	EPA 625	Pass		
Base Neutral Extractables	4200	5545	Aniline	EPA 625	Pass		
WP Base/Neutrals	711	5545	Aniline	EPA 625	Pass		
Base Neutral Extractables	4200	5555	Anthracene	EPA 625	Pass		
PAH-GC & GCMS	4880	5555	Anthracene	EPA 625	Pass		
WP Base/Neutrals	711	5555	Anthracene	EPA 625	Pass		
Base Neutral Extractables	4200	5595	Benzidine	EPA 625	Pass		
WP Base/Neutrals	711	5595	Benzidine	EPA 625	Pass		
Base Neutral Extractables	4200	5575	Benzo(a)anthracene	EPA 625	Pass		
PAH-GC & GCMS	4880	5575	Benzo(a)anthracene	EPA 625	Pass		
WP Base/Neutrals	711	5575	Benzo(a)anthracene	EPA 625	Pass		
Base Neutral Extractables	4200	5580	Benzo(a)pyrene	EPA 625	Pass		
PAH-GC & GCMS	4880	5580	Benzo(a)pyrene	EPA 625	Pass		
WP Base/Neutrals	711	5580	Benzo(a)pyrene	EPA 625	Pass		
Base Neutral Extractables	4200	5585	Benzo(b)fluoranthene	EPA 625	Pass		
PAH-GC & GCMS	4880	5585	Benzo(b)fluoranthene	EPA 625	Pass		
WP Base/Neutrals	711	5585	Benzo(b)fluoranthene	EPA 625	Pass		
Base Neutral Extractables	4200	5590	Benzo(g,h,i)perylene	EPA 625	Pass		
PAH-GC & GCMS	4880	5590	Benzo(g,h,i)perylene	EPA 625	Pass		
WP Base/Neutrals	711	5590	Benzo(g,h,i)perylene	EPA 625	Pass		
Base Neutral Extractables	4200	5600	Benzo(k)fluoranthene	EPA 625	Pass		
PAH-GC & GCMS	4880	5600	Benzo(k)fluoranthene	EPA 625	Pass		
WP Base/Neutrals	711	5600	Benzo(k)fluoranthene	EPA 625	Pass		
Acid Extractables	4190	5610	Benzoic Acid	EPA 625	Pass		
Acids	712	5610	Benzoic acid	EPA 625	Pass		
Base Neutral Extractables	4200	5630	Benzyl alcohol	EPA 625	Pass		
WP Base/Neutrals	711	5630	Benzyl alcohol	EPA 625	Pass		
Base Neutral Extractables	4200	5670	Benzyl butyl phthalate	EPA 625	Pass		
Base Neutral Extractables	4200	5760	bis(2-Chloroethoxy) methane	EPA 625	Pass		
WP Base/Neutrals	711	5760	bis(2-Chloroethoxy) methane	EPA 625	Pass		
Base Neutral Extractables	4200	5765	bis(2-Chloroethyl) ether	EPA 625	Pass		
WP Base/Neutrals	711	5765	bis(2-Chloroethyl)ether	EPA 625	Pass		
Base Neutrals Extractables	4200	5780	bis(2-Chloroisopropyl) ether	EPA 625	Pass		
WP Base/Neutrals	711	5780	bis(2-Chloroisopropyl) ether	EPA 625	Pass		
Base Neutral Extractables	4200	6255	bis(2-Ethylhexyl) phthalate	EPA 625	Pass		
WP Base/Neutrals	711	6255	bis(2-Ethylhexyl) phthalate	EPA 625	Pass		
WP Base/Neutrals	711	5670	Butylbenzylphthalate	EPA 625	Pass		
Base Neutral Extractables	4200	5680	Carbazole	EPA 625	Pass		
WP Base/Neutrals	711	5680	Carbazole	EPA 625	Pass		
Base Neutral Extractables	4200	5855	Chrysene	EPA 625	Pass		
PAH-GC & GCMS	4880	5855	Chrysene	EPA 625	Pass		
WP Base/Neutrals	711	5855	Chrysene	EPA 625	Pass		
Base Neutrals Extractables	4200	5895	Dibenz(a,h) anthracene	EPA 625	Pass		
PAH-GC & GCMS	4880	5895	Dibenz(a,h) anthracene	EPA 625	Pass		
WP Base/Neutrals	711	5895	Dibenz(a,h) anthracene	EPA 625	Pass		
Base Neutral Extractables	4200	5905	Dibenzofuran	EPA 625	Pass		
WP Base/Neutrals	711	5905	Dibenzofuran	EPA 625	Pass		
Base Neutral Extractables	4200	6070	Diethyl phthalate	EPA 625	Pass		
WP Base/Neutrals	711	6070	Diethylphthalate	EPA 625	Pass		
Base Neutral Extractables	4200	6135	Dimethyl phthalate	EPA 625	Pass		
WP Base/Neutrals	711	6135	Dimethyl phthalate	EPA 625	Pass		
Base Neutral Extractables	4200	5925	Di-n-butylphthalate	EPA 625	Pass		
WP Base/Neutrals	711	5925	Di-n-butylphthalate	EPA 625	Pass		
Base Neutral Extractables	4200	6200	Di-n-octylphthalate	EPA 625	Pass		
WP Base/Neutrals	711	6200	Di-n-octylphthalate	EPA 625	Pass		
Base Neutral Extractables	4200	6265	Fluoranthene	EPA 625	Pass		
PAH-GC & GCMS	4880	6265	Fluoranthene	EPA 625	Pass		
WP Base/Neutrals	711	6265	Fluoranthene	EPA 625	Pass		
Base Neutral Extractables	4200	6270	Fluorene	EPA 625	Pass		
PAH-GC & GCMS	4880	6270	Fluorene	EPA 625	Pass		
WP Base/Neutrals	711	6270	Fluorene	EPA 625	Pass		
Base Neutral Extractables	4200	6275	Hexachlorobenzene	EPA 625	Pass		
WP Base/Neutrals	711	6275	Hexachlorobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	4835	Hexachlorobutadiene	EPA 625	Pass		
WP Base/Neutrals	711	4835	Hexachlorobutadiene	EPA 625	Pass		
Base Neutral Extractables	4200	6285	Hexachlorocyclopentadiene	EPA 625	Pass		
WP Base/Neutrals	711	6285	Hexachlorocyclopentadiene	EPA 625	Pass		
Base Neutral Extractables	4200	4840	Hexachloroethane	EPA 625	Pass		
WP Base/Neutrals	711	4840	Hexachloroethane	EPA 625	Pass		
Base Neutral Extractables	4200	6315	Indeno (1,2,3-cd) pyrene	EPA 625	Pass		
PAH-GC & GCMS	4880	6315	Indeno (1,2,3-cd) pyrene	EPA 625	Pass		
WP Base/Neutrals	711	6315	Indeno (1,2,3-cd) pyrene	EPA 625	Pass		
WP Base/Neutrals	711	6320	Isophorone	EPA 625	Pass		
Base Neutral Extractables	4200	6320	Isophorone	EPA 625	Pass		
Base Neutral Extractables	4200	5005	Naphthalene	EPA 625	Pass		
PAH-GC & GCMS	4880	5005	Naphthalene	EPA 625	Pass		
WP Base/Neutrals	711	5005	Naphthalene	EPA 625	Pass		
WP Base/Neutrals	711	5015	Nitrobenzene	EPA 625	Pass		
Base Neutral Extractables	4200	5015	Nitrobenzene (NB)	EPA 625	Pass		
Base Neutral Extractables	4200	6530	N-nitrosodimethylamine	EPA 625	Pass		
WP Base/Neutrals	711	6530	N-Nitrosodimethylamine	EPA 625	Pass		
Base Neutral Extractables	4200	6545	N-Nitroso-di-n-propylamine	EPA 625	Pass		
WP Base/Neutrals	711	6545	N-Nitroso-di-n-propylamine	EPA 625	Pass		
Base Neutral Extractables	4200	6535	N-nitrosodiphenylamine	EPA 625	Pass		
WP Base/Neutrals	711	6535	N-Nitrosodiphenylamine	EPA 625	Pass		
Acid Extractables	4190	6605	Pentachlorophenol	EPA 625	Pass		
Acids	712	6605	Pentachlorophenol	EPA 625	Pass		
Acid Extractables	4190	6605	Pentachlorophenol	EPA 625	Pass		
Base Neutral Extractables	4200	6615	Phenanthrene	EPA 625	Pass		
PAH-GC & GCMS	4880	6615	Phenanthrene	EPA 625	Pass		

WP Base/Neutrals	711	6615	Phenanthrene	EPA 625	Pass		
Acid Extractables	4190	6625	Phenol	EPA 625	Pass		
Acids	712	6625	Phenol	EPA 625	Pass		
Base Neutral Extractables	4200	6665	Pyrene	EPA 625	Pass		
PAH-GC & GCMS	4880	6665	Pyrene	EPA 625	Pass		
WP Base/Neutrals	711	6665	Pyrene	EPA 625	Pass		
Base Neutral Extractables	4200	5095	Pyridine	EPA 625	Pass		
WP Base/Neutrals	711	5095	Pyridine	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5500	Acenaphthene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5505	Acenaphthylene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5555	Anthracene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5575	Benzo(a)anthracene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5580	Benzo(a)pyrene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5585	Benzo(b)fluoranthene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5601	Benzo(b+k)fluoranthene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5590	Benzo(g,h,i)perylene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5600	Benzo(k)fluoranthene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5855	Chrysene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5895	Dibenz(a,h) anthracene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	6265	Fluoranthene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	6270	Fluorene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	6315	Indeno (1,2,3-cd) pyrene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	5005	Naphthalene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	6615	Phenanthrene	EPA 625	Pass		
Base/Neutrals 1	PEO-121-1	6665	Pyrene	EPA 625	Pass		
WP Perchlorate	55116	1895	Perchlorate	EPA 6850	Pass		
WP Hexavalent Chromium	55096	1045	Chromium VI	EPA 7196A	Pass		
Hexavalent Chromium	4180	1045	Chromium, Hexavalent	EPA 7196A	Pass		
WP Hexavalent Chromium	55096	1045	Chromium VI	EPA 7199	Pass		
Hexavalent Chromium	4180	1045	Chromium, Hexavalent	EPA 7199	Pass		
Trace Metals	4070	1095	Mercury	EPA 7470A	Pass		
WP & DMRQA Trace Elements	55024	1095	Mercury	EPA 7470A	Pass		
Trace Metals 1	PEI-034-1	1095	Mercury, Hg	EPA 7470A	Pass		
PT Diesel Fuel #2 in Water	38114	9369	#2 Fuel Oil (Diesel)	EPA 8015B	Pass		
Diesel Range Organics (DRO)	4830	9369	Diesel Range Organics (DRO)	EPA 8015B	Pass		
Gasoline Range Organics (GRO)	4840	9408	Gasoline Range Organics	EPA 8015B	Pass		
PT Unleaded Gasoline in Water	38116	9408	Unleaded Gasoline 93 Octane	EPA 8015B	Pass		
BTEX	4230	4375	Benzene	EPA 8021B	Pass		
BTEX & MTBE in Water	643	4375	Benzene	EPA 8021B	Pass		
BTEX	4230	4765	Ethylbenzene	EPA 8021B	Pass		
BTEX & MTBE in Water	643	4765	Ethylbenzene	EPA 8021B	Pass		
BTEX	4230	5000	Methyl-t-butylether (MTBE)	EPA 8021B	Pass		
BTEX & MTBE in Water	643	5000	tert-Butyl methyl ether (MTBE)	EPA 8021B	Pass		
BTEX	4230	5140	Toluene	EPA 8021B	Pass		
BTEX & MTBE in Water	643	5140	Toluene	EPA 8021B	Pass		
BTEX	4230	5260	Total Xylenes	EPA 8021B	Pass		
BTEX & MTBE in Water	643	5260	Xylenes, total	EPA 8021B	Pass		
Pesticides (WP)	4460	7355	4,4'-DDD	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7355	4,4'-DDD	EPA 8081A	Pass		
Pesticides (WP)	4460	7360	4,4'-DDE	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7360	4,4'-DDE	EPA 8081A	Pass		
Pesticides (WP)	4460	7365	4,4'-DDT	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7365	4,4'-DDT	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7810	4,4'-Methoxychlor	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7110	a-BHC	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7240	a-Chlordane	EPA 8081A	Pass		
Pesticides (WP)	4460	7025	Aldrin	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7025	Aldrin	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7110	alpha-BHC	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7240	alpha-Chlordane	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7115	b-BHC	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7115	beta-BHC	EPA 8081A	Pass		
WP Pesticide Amp 3	38047	7250	Chlordane (total)	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7105	d-BHC	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7105	delta-BHC	EPA 8081A	Pass		
Pesticides (WP)	4460	7470	Dieldrin	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7470	Dieldrin	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7510	Endosulfan I	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7515	Endosulfan II	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7515	Endosulfan II	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7520	Endosulfan sulfate	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7520	Endosulfan sulfate	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7540	Endrin	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7540	Endrin	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7530	Endrin aldehyde	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7530	Endrin aldehyde	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7535	Endrin Ketone	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7535	Endrin ketone	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7120	gamma-BHC (Lindane)	EPA 8081A	Pass		
Pesticides (NELAC)	4470	7120	gamma-BHC (Lindane)	EPA 8081A	Pass		
Pesticides (NELAC)	4470	7245	gamma-Chlordane	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7245	gamms-Chlordane	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7120	g-BHC (Lindane)	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7245	g-Chlordane	EPA 8081A	Pass		
Pesticides (WP)	4460	7685	Heptachlor	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7685	Heptachlor	EPA 8081A	Pass		
Pesticides (WP)	4460	7690	Heptachlor epoxide	EPA 8081A	Pass		
WP Organochlorine Pesticides	38122	7690	Heptachlor epoxide	EPA 8081A	Pass		
Pesticides (NELAC)	4460	7810	Methoxychlor	EPA 8081A	Pass		
Total Chlordane	4160	7250	Total Chlordane	EPA 8081A	Pass		
Toxaphene	4270	8250	Toxaphene	EPA 8081A	Pass		

WP Acrolein & Acrylonitrile	38123	8250	Toxaphene	EPA 8081A	Pass		
WP PCBs in Water	38091	8880	Aroclor 1016	EPA 8082	Pass		
WP PCBs in Water	38094	8880	Aroclor 1016	EPA 8082	Pass		
WP PCBs in Water	832S	8880	Aroclor 1016	EPA 8082	Pass		
PCBs in Oil	4140	8880	Aroclor 1016 in Oil	EPA 8082	Pass		
PCBs in Water	4130	8880	Aroclor 1016 Sample 1	EPA 8082	Pass		
PCBs in Water	4130	8880	Aroclor 1016 Sample 2	EPA 8082	Pass		
PCBs in Water	PEO-020	8912	Aroclor 1016/1242	EPA 8082	Pass		
PCBs in Water	832S	8885	Aroclor 1221	EPA 8082	Pass		
WP PCBs in Water	38091	8885	Aroclor 1221	EPA 8082	Pass		
WP PCBs in Water	38094	8885	Aroclor 1221	EPA 8082	Pass		
PCBs in Oil	4140	8885	Aroclor 1221 in Oil	EPA 8082	Pass		
WP PCBs in Water	38091	8890	Aroclor 1232	EPA 8082	Pass		
WP PCBs in Water	38094	8890	Aroclor 1232	EPA 8082	Pass		
WP PCBs in Water	832S	8890	Aroclor 1232	EPA 8082	Pass		
PCBs in Oil	4140	8890	Aroclor 1232 in Oil	EPA 8082	Pass		
PCBs in Water	4130	8890	Aroclor 1232 Sample 1	EPA 8082	Pass		
WP PCBs in Water	832S	8895	Aroclor 1242	EPA 8082	Pass		
WP PCBs in Water	38091	8895	Aroclor 1242	EPA 8082	Pass		
WP PCBs in Water	38094	8895	Aroclor 1242	EPA 8082	Pass		
PCBs in Oil	4140	8895	Aroclor 1242 in Oil	EPA 8082	Pass		
PCBs in Water	4130	8895	Aroclor 1242 Sample 1	EPA 8082	Pass		
PCBs in Water	4130	8895	Aroclor 1242 Sample 2	EPA 8082	Pass		
WP PCBs in Water	38091	8900	Aroclor 1248	EPA 8082	Pass		
WP PCBs in Water	38094	8900	Aroclor 1248	EPA 8082	Pass		
PCBs in Oil	4140	8900	Aroclor 1248 in Oil	EPA 8082	Pass		
PCBs in Water	4130	8900	Aroclor 1248 Sample 1	EPA 8082	Pass		
PCBs in Water	4130	8900	Aroclor 1248 Sample 2	EPA 8082	Pass		
WP PCBs in Water	38091	8905	Aroclor 1254	EPA 8082	Pass		
WP PCBs in Water	38094	8905	Aroclor 1254	EPA 8082	Pass		
PCBs in Oil	4140	8905	Aroclor 1254 in Oil	EPA 8082	Pass		
PCBs in Water	4130	8905	Aroclor 1254 Sample 1	EPA 8082	Pass		
PCBs in Water	4130	8905	Aroclor 1254 Sample 2	EPA 8082	Pass		
WP PCBs in Water	38091	8910	Aroclor 1260	EPA 8082	Pass		
WP PCBs in Water	38094	8910	Aroclor 1260	EPA 8082	Pass		
WP PCBs in Water	832S	8910	Aroclor 1260	EPA 8082	Pass		
PCBs in Water	4130	8910	Aroclor 1260 Sample 1	EPA 8082	Pass		
PCBs in Water	4130	8910	Aroclor 1260 Sample 2	EPA 8082	Pass		
PCBs in Water	PEO-020	8880	Aroclor-1016 (PCB-1016)	EPA 8082	Pass		
PCBs in Water	PEO-020	8885	Aroclor-1221 (PCB-1221)	EPA 8082	Pass		
PCBs in Water	PEO-020	8890	Aroclor-1232 (PCB-1232)	EPA 8082	Pass		
PCBs in Water	PEO-020	8895	Aroclor-1242 (PCB-1242)	EPA 8082	Pass		
PCBs in Water	PEO-020	8900	Aroclor-1248 (PCB-1248)	EPA 8082	Pass		
PCBs in Water	PEO-020	8905	Aroclor-1254 (PCB-1254)	EPA 8082	Pass		
PCBs in Water	PEO-020	8910	Aroclor-1260 (PCB-1260)	EPA 8082	Pass		
WP PCBs in Transformer Oil	38092	8880	PCB in Oil 1016 or 1242	EPA 8082	Pass		
WP PCBs in Water	38094	8880	PCB in Oil 1016 or 1242	EPA 8082	Pass		
WP PCBs in Transformer Oil	38092	8905	PCB in Oil 1254	EPA 8082	Pass		
WP PCBs in Water	38094	8905	PCB in Oil 1254	EPA 8082	Pass		
WP PCBs in Transformer Oil	38092	8910	PCB in Oil 1260	EPA 8082	Pass		
WP PCBs in Water	38094	8910	PCB in Oil 1260	EPA 8082	Pass		
OP Pesticides/Herbicides	4810	7075	Azinphos-methyl	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7075	Azinphosmethyl (Guthion)	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7300	Chlorpyrifos	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7390	Demeton O&S	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7390	Demeton, (Mix of Isomers O:S [35%:56%])	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7410	Diazinon	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7410	Diazinon	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	8610	Dichlorvos (DDVP)	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7475	Dimethoate	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	8625	Disulfoton	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	8625	Disulfoton	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7565	Ethion	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7565	Ethion	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7570	Ethoprop	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7580	Famphur	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7770	Malathion	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7770	Malathion	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7955	Parathion ethyl	EPA 8141A	Pass		
WP Organophosphorous Pesticides	38135	7825	Parathion methyl	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7955	Parathion, ethyl	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7825	Parathion, methyl	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	7985	Phorate	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	8000	Phosmet	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	8110	Ronnel	EPA 8141A	Pass		
OP Pesticides/Herbicides	4810	8200	Stirophos (Tetrachlorvinphos)	EPA 8141A	Pass		
Herbicides	4440	8655	2,4,5-T	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	8655	2,4,5-T	EPA 8151A	Pass		
Herbicides	4440	8650	2,4,5-TP (Silvex)	EPA 8151A	Pass		
Herbicides	4440	8545	2,4-D	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8545	2,4-D (2,4-Dichlorophenoxyacetic acid)	EPA 8151A	Pass		
Herbicides	4440	8560	2,4-DB	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	8560	2,4-DB	EPA 8151A	Pass		
Herbicides	4440	8600	3,5-Dichlorobenzoic acid	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	8600	3,5-Dichlorobenzoic acid	EPA 8151A	Pass		
Herbicides	4440	6500	4-Nitrophenol	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	6500	4-Nitrophenol	EPA 8151A	Pass		
Herbicides	4440	8505	Acifluorfen	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8505	Acifluorfen	EPA 8151A	Pass		
Herbicides	4440	8530	Bentazon	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	8530	Bentazon	EPA 8151A	Pass		

Herbicides	4440	8540	Chloramben	EPA 8151A	Pass		
Herbicides	4440	8550	Dacthal diacid (DCPA)	EPA 8151A	Pass		
Herbicides	4440	8555	Dalapon	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8555	Dalapon	EPA 8151A	Pass		
Herbicides	4440	8595	Dicamba	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8595	Dicamba	EPA 8151A	Pass		
Herbicides	4440	8605	Dichloroprop	EPA 8151A	Pass		
WP Herbicide Acid Mix #2	38136	8605	Dichloroprop	EPA 8151A	Pass		
Herbicides	4440	8620	Dinoseb	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8620	Dinoseb (2-sec-Butyl-4,6-dinitrophenol)	EPA 8151A	Pass		
Herbicides	4440	7775	MCPA	EPA 8151A	Pass		
Herbicides	4440	7780	MCPP	EPA 8151A	Pass		
Herbicides	4440	6605	Pentachlorophenol	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	6605	Pentachlorophenol	EPA 8151A	Pass		
Herbicides	4440	8645	Picloram	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8645	Picloram	EPA 8151A	Pass		
WP Acrolein & Acrylonitrile	38123	8650	Silvex (2,4,5-TP)	EPA 8151A	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass		
Volatiles	4170	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass		
Volatile Organic Compounds 2	PEO-120-2	5160	1,1,1-Trichloroethane	EPA 8260B	Pass		
Volatiles	4170	5160	1,1,1-Trichloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5160	1,1,1-Trichloroethane	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass		
Volatiles	4170	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	5165	1,1,2-Trichloroethane	EPA 8260B	Pass		
Volatiles	4170	5165	1,1,2-Trichloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5165	1,1,2-Trichloroethane	EPA 8260B	Pass		
WP Oxygenates	38157	5185	1,1,2-Trichlorotrifluoroethane	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4630	1,1-Dichloroethane	EPA 8260B	Pass		
Volatiles	4170	4630	1,1-Dichloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4630	1,1-Dichloroethane	EPA 8260B	Pass		
Volatiles	4170	4640	1,1-Dichloroethene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4640	1,1-Dichloroethene	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4640	1,1-Dichloroethylene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4670	1,1-Dichloropropene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5150	1,2,3-Trichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	5180	1,2,3-Trichloropropane	EPA 8260B	Pass		
Volatiles	4170	5180	1,2,3-Trichloropropane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5180	1,2,3-Trichloropropane	EPA 8260B	Pass		
Volatiles	4170	5155	1,2,4-Trichlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5155	1,2,4-Trichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	5210	1,2,4-Trimethylbenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5210	1,2,4-Trimethylbenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4570	1,2-Dibromo-3-chloropropane	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4570	1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260B	Pass		
Volatiles	4170	4570	1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260B	Pass		
Volatiles	4170	4585	1,2-Dibromoethane (EDB)	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4585	1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260B	Pass		
Volatile Aromatics	4450	4610	1,2-Dichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	4610	1,2-Dichlorobenzene	EPA 8260B	Pass		
Volatiles	4170	4610	1,2-Dichlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4610	1,2-Dichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 2	PEO-120-2	4635	1,2-Dichloroethane	EPA 8260B	Pass		
Volatiles	4170	4635	1,2-Dichloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4635	1,2-Dichloroethane	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4655	1,2-Dichloropropane	EPA 8260B	Pass		
Volatiles	4170	4655	1,2-Dichloropropane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4655	1,2-Dichloropropane	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	5215	1,3,5-Trimethylbenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5215	1,3,5-Trimethylbenzene	EPA 8260B	Pass		
Volatile Aromatics	4450	4615	1,3-Dichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	4615	1,3-Dichlorobenzene	EPA 8260B	Pass		
Volatiles	4170	4615	1,3-Dichlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4615	1,3-Dichlorobenzene	EPA 8260B	Pass		
Volatile Aromatics	4450	4620	1,4-Dichlorobenzene	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	4620	1,4-Dichlorobenzene	EPA 8260B	Pass		
Volatiles	4170	4620	1,4-Dichlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4620	1,4-Dichlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4665	2,2-Dichloropropane	EPA 8260B	Pass		
WP Ketones	38134	4410	2-Butanone	EPA 8260B	Pass		
Volatiles	4170	4410	2-Butanone (Methyl ethyl ketone)	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4410	2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4500	2-Chloroethyl vinyl ether	EPA 8260B	Pass		
Volatiles	4170	4500	2-Chloroethyl vinyl ether	EPA 8260B	Pass		
WP 2-Chloroethyl vinyl ether	38128	4500	2-Chloroethyl vinyl ether	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4535	2-Chlorotoluene	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4860	2-Hexanone	EPA 8260B	Pass		
Volatiles	4170	4860	2-Hexanone	EPA 8260B	Pass		
WP Ketones	38134	4860	2-Hexanone	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4540	4-Chlorotoluene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4995	4-Methyl-2-pentanone	EPA 8260B	Pass		
WP Ketones	38134	4995	4-Methyl-2-pentanone	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4995	4-Methyl-2-pentanone (MIBK)	EPA 8260B	Pass		
Volatiles	4170	4995	4-Methyl-2-pentanone (MIBK)	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4315	Acetone	EPA 8260B	Pass		
Volatiles	4170	4315	Acetone	EPA 8260B	Pass		
WP Ketones	38134	4315	Acetone	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	4320	Acetonitrile	EPA 8260B	Pass		
Volatiles	4170	4325	Acrolein	EPA 8260B	Pass		

WP Acrolein & Acrylonitrile	38123	4325	Acrolein	EPA 8260B	Pass		
Volatiles Organic Compounds 3B	PEO-120-3B	4325	Acrolein (Propenal)	EPA 8260B	Pass		
Volatiles Organic Compounds 3B	PEO-120-3B	4340	Acrylonitrile	EPA 8260B	Pass		
Volatiles	4170	4340	Acrylonitrile	EPA 8260B	Pass		
WP Acrolein & Acrylonitrile	38123	4340	Acrylonitrile	EPA 8260B	Pass		
CWA BTEX & MTBE	38166	4375	Benzene	EPA 8260B	Pass		
Volatiles Aromatics	4450	4375	Benzene	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	4375	Benzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4375	Benzene	EPA 8260B	Pass		
Volatiles	4170	4375	Beznene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4385	Bromobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4390	Bromochloromethane	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4395	Bromodichloromethane	EPA 8260B	Pass		
Volatiles	4170	4395	Bromodichloromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4395	Bromodichloromethane	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4400	Bromoform	EPA 8260B	Pass		
Volatiles	4170	4400	Bromoform	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4400	Bromoform	EPA 8260B	Pass		
Volatiles	4170	4950	Bromomethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4950	Bromomethane	EPA 8260B	Pass		
Volatiles	4170	4450	Carbon disulfide	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4450	Carbon disulphide	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4455	Carbon tetrachloride	EPA 8260B	Pass		
Volatiles	4170	4455	Carbon tetrachloride	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4455	Carbon tetrachloride	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4475	Chlorobenzene	EPA 8260B	Pass		
Volatiles	4170	4475	Chlorobenzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4475	Chlorobenzene	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	4485	Chloroethane	EPA 8260B	Pass		
Volatiles	4170	4485	Chloroethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4485	Chloroethane	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4505	Chloroform	EPA 8260B	Pass		
Volatiles	4170	4505	Chloroform	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4505	Chloroform	EPA 8260B	Pass		
Volatiles	4170	4960	Chloromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4960	Chloromethane	EPA 8260B	Pass		
Volatiles	4170	4645	cis-1,2-Dichloroethene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4645	cis-1,2-Dichloroethene	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	4645	cis-1,2-Dichloroethylene	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles	4170	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4575	Dibromochloromethane	EPA 8260B	Pass		
Volatiles	4170	4575	Dibromochloromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4575	Dibromochloromethane	EPA 8260B	Pass		
Volatiles Organic Compounds 3B	PEO-120-3B	4595	Dibromomethane	EPA 8260B	Pass		
Volatiles	4170	4595	Dibromomethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4595	Dibromomethane	EPA 8260B	Pass		
Volatiles	4170	4625	Dichlorodifluoromethane	EPA 8260B	Pass		
Volatiles Organic Compounds 3B	PEO-120-3B	4625	Dichlorodifluoromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4625	Dichlorodifluoromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4765	Ethyl benzene	EPA 8260B	Pass		
CWA BTEX & MTBE	38166	4765	Ethylbenzene	EPA 8260B	Pass		
Volatiles Aromatics	4450	4765	Ethylbenzene	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	4765	Ethylbenzene	EPA 8260B	Pass		
Volatiles	4170	4765	Ethylbenzene	EPA 8260B	Pass		
Volatiles	4170	4835	Hexachlorobutadiene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4835	Hexachlorobutadiene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4840	Hexachloroethane	EPA 8260B	Pass		
WP Oxygenates	38157	9375	Isopropyl ether (DIPE)	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4900	Isopropylbenzene	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	5240	m+p-Xylene	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	4950	Methyl bromide (Bromomethane)	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	4960	Methyl chloride (Chloromethane)	EPA 8260B	Pass		
CWA BTEX & MTBE	38166	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass		
WP Oxygenates	38157	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4975	Methylene chloride	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	4975	Methylene chloride (Dichloromethane)	EPA 8260B	Pass		
Volatiles	4170	4975	Methylene chloride (Dichloromethane)	EPA 8260B	Pass		
Volatiles	4170	5000	Methyl-t-butylether (MTBE)	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	5005	Naphthalene	EPA 8260B	Pass		
Volatiles	4170	5005	Naphthalene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5005	Naphthalene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4435	n-Butyl benzene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5090	n-Propylbenzene	EPA 8260B	Pass		
WP Oxygenates	38157	5090	n-Propylbenzene	EPA 8260B	Pass		
Volatiles Organic Compounds 1	PEO-120-1	5250	o-Xylene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4910	p-Isopropyl toluene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4440	sec-Butyl benzene	EPA 8260B	Pass		
Volatiles Organic Compounds 3A	PEO-120-3A	5100	Styrene	EPA 8260B	Pass		
Volatiles	4170	5100	Styrene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5100	Styrene	EPA 8260B	Pass		
WP Oxygenates	38157	4370	tert-Amyl methyl ether (TAME)	EPA 8260B	Pass		
WP Oxygenates	38157	4420	tert-Butyl alcohol (t-Butanol)	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4445	tert-Butyl benzene	EPA 8260B	Pass		
WP Oxygenates	38157	4770	tert-Butyl ethyl ether (ETBE)	EPA 8260B	Pass		
Volatiles	4170	5115	Tetrachloroethene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5115	Tetrachloroethene	EPA 8260B	Pass		
Volatiles Organic Compounds 2	PEO-120-2	5115	Tetrachloroethylene (Perchloroethylene)	EPA 8260B	Pass		

CWA BTEX & MTBE	38166	5140	Toluene	EPA 8260B	Pass		
Volatile Aromatics	4450	5140	Toluene	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	5140	Toluene	EPA 8260B	Pass		
Volatiles	4170	5140	Toluene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5140	Toluene	EPA 8260B	Pass		
CWA BTEX & MTBE	38166	5260	Total Xylenes	EPA 8260B	Pass		
Volatile Aromatics	4450	5260	Total Xylenes	EPA 8260B	Pass		
Volatiles	4170	5260	Total Xylenes	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5260	Total Xylenes	EPA 8260B	Pass		
Volatiles	4170	4700	trans-1,2-Dichloroethene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4700	trans-1,2-Dichloroethene	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4700	trans-1,2-Dichloroethylene	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles	4170	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass		
Volatiles	4170	5170	Trichloroethene	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5170	Trichloroethene	EPA 8260B	Pass		
Volatile Organic Compounds 2	PEO-120-2	5170	Trichloroethene (Trichloroethylene)	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	5175	Trichlorofluoromethane	EPA 8260B	Pass		
Volatiles	4170	5175	Trichlorofluoromethane	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5175	Trichlorofluoromethane	EPA 8260B	Pass		
Volatile Organic Compounds 3B	PEO-120-3B	5225	Vinyl acetate	EPA 8260B	Pass		
Volatiles	4170	5225	Vinyl acetate	EPA 8260B	Pass		
Volatile Organic Compounds 3A	PEO-120-3A	5235	Vinyl chloride	EPA 8260B	Pass		
Volatiles	4170	5235	Vinyl chloride	EPA 8260B	Pass		
Volatiles in Non-Potable Water	38083	5235	Vinyl chloride	EPA 8260B	Pass		
Volatile Organic Compounds 1	PEO-120-1	5260	Xylene, total	EPA 8260B	Pass		
Base Neutral Extractables	4200	6715	1,2,4,5-Tetrachlorobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5155	1,2,4-Trichlorobenzene	EPA 8270C	Pass		
WP Base/Neutrals	711	5155	1,2,4-Trichlorobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	4610	1,2-Dichlorobenzene	EPA 8270C	Pass		
WP Base/Neutrals	711	4610	1,2-Dichlorobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	4615	1,3-Dichlorobenzene	EPA 8270C	Pass		
WP Base/Neutrals	711	4615	1,3-Dichlorobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	4620	1,4-Dichlorobenzene	EPA 8270C	Pass		
WP Base/Neutrals	711	4620	1,4-Dichlorobenzene	EPA 8270C	Pass		
Acid Extractables	4190	6735	2,3,4,6-Tetrachlorophenol	EPA 8270C	Pass		
Acids	712	6735	2,3,4,6-Tetrachlorophenol	EPA 8270C	Pass		
Acid Extractables	4190	6835	2,4,5-Trichlorophenol	EPA 8270C	Pass		
Acids	712	6835	2,4,5-Trichlorophenol	EPA 8270C	Pass		
Acids	712	6840	2,4,6-Trichlorophenol	EPA 8270C	Pass		
Acid Extractables	4190	6840	2,4,6-Trichlorophenol	EPA 8270C	Pass		
Acid Extractables	4190	6000	2,4-Dichlorophenol	EPA 8270C	Pass		
Acids	712	6000	2,4-Dichlorophenol	EPA 8270C	Pass		
Acid Extractables	4190	6130	2,4-Dimethylphenol	EPA 8270C	Pass		
Acids	712	6130	2,4-Dimethylphenol	EPA 8270C	Pass		
Acid Extractables	4190	6175	2,4-Dinitrophenol	EPA 8270C	Pass		
Acids	712	6175	2,4-Dinitrophenol	EPA 8270C	Pass		
WP Base/Neutrals	711	6185	2,4-Dinitrotoluene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 8270C	Pass		
Acid Extractables	4190	6005	2,6-Dichlorophenol	EPA 8270C	Pass		
Acids	712	6005	2,6-Dichlorophenol	EPA 8270C	Pass		
WP Base/Neutrals	711	6190	2,6-Dinitrotoluene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6190	2,6-Dinitrotoluene (2,6-DNT)	EPA 8270C	Pass		
Base Neutral Extractables	4200	5795	2-Chloronaphthalene	EPA 8270C	Pass		
WP Base/Neutrals	711	5795	2-Chloronaphthalene	EPA 8270C	Pass		
Acid Extractables	4190	5800	2-Chlorophenol	EPA 8270C	Pass		
Acids	712	5800	2-Chlorophenol	EPA 8270C	Pass		
Acid Extractables	4190	6360	2-Methyl-4,6-Dinitrophenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	6385	2-Methylnaphthalene	EPA 8270C	Pass		
WP Base/Neutrals	711	6385	2-Methylnaphthalene	EPA 8270C	Pass		
Acid Extractables	4190	6400	2-Methylphenol	EPA 8270C	Pass		
Acids	712	6400	2-Methylphenol	EPA 8270C	Pass		
WP Base/Neutrals	711	6460	2-Nitroaniline	EPA 8270C	Pass		
Base Neutral Extractables	4200	6460	2-Nitroaniline	EPA 8270C	Pass		
Acid Extractables	4190	6490	2-Nitrophenol	EPA 8270C	Pass		
Acids	712	6490	2-Nitrophenol	EPA 8270C	Pass		
Acid Extractables	4190	6410	3 & 4-Methylphenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	5945	3,3'-Dichlorobenzidine	EPA 8270C	Pass		
WP Base/Neutrals	711	5945	3,3'-Dichlorobenzidine	EPA 8270C	Pass		
Acid Extractables	4190	6405	3-Methylphenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	6465	3-Nitroaniline	EPA 8270C	Pass		
WP Base/Neutrals	711	6465	3-Nitroaniline	EPA 8270C	Pass		
Acids	712	6360	4,6-Dinitro-2-methylphenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	5660	4-Bromophenyl phenyl ether	EPA 8270C	Pass		
WP Base/Neutrals	711	5660	4-Bromophenyl-phenylether	EPA 8270C	Pass		
Acid Extractables	4190	5700	4-Chloro-3-methylphenol	EPA 8270C	Pass		
Acids	712	5700	4-Chloro-3-methylphenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	5745	4-Chloroaniline	EPA 8270C	Pass		
WP Base/Neutrals	711	5745	4-Chloroaniline	EPA 8270C	Pass		
Base Neutral Extractables	4200	5825	4-Chlorophenyl-phenylether	EPA 8270C	Pass		
WP Base/Neutrals	711	5825	4-Chlorophenyl-phenylether	EPA 8270C	Pass		
Acids	712	6410	4-Methylphenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	6470	4-Nitroaniline	EPA 8270C	Pass		
WP Base/Neutrals	711	6470	4-Nitroaniline	EPA 8270C	Pass		
Acid Extractables	4190	6500	4-Nitrophenol	EPA 8270C	Pass		
Acids	712	6500	4-Nitrophenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	5500	Acenaphthene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5500	Acenaphthene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5500	Acenaphthene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5500	Acenaphthene	EPA 8270C	Pass		

WP Base/Neutrals	711	5500	Acenaphthene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5505	Acenaphthylene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5505	Acenaphthylene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5505	Acenaphthylene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5505	Acenaphthylene	EPA 8270C	Pass		
WP Base/Neutrals	711	5505	Acenaphthylene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5545	Aniline	EPA 8270C	Pass		
WP Base/Neutrals	711	5545	Aniline	EPA 8270C	Pass		
Base Neutral Extractables	4200	5555	Anthracene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5555	Anthracene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5555	Anthracene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5555	Anthracene	EPA 8270C	Pass		
WP Base/Neutrals	711	5555	Anthracene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5595	Benzidine	EPA 8270C	Pass		
WP Base/Neutrals	711	5595	Benzidine	EPA 8270C	Pass		
Base Neutral Extractables	4200	5575	Benzo(a)anthracene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5575	Benzo(a)anthracene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5575	Benzo(a)anthracene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5575	Benzo(a)anthracene	EPA 8270C	Pass		
WP Base/Neutrals	711	5575	Benzo(a)anthracene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5580	Benzo(a)pyrene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5580	Benzo(a)pyrene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5580	Benzo(a)pyrene	EPA 8270C	Pass		
WP Base/Neutrals	711	5580	Benzo(a)pyrene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5585	Benzo(b)fluoranthene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5585	Benzo(b)fluoranthene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5585	Benzo(b)fluoranthene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5585	Benzo(b)fluoranthene	EPA 8270C	Pass		
WP Base/Neutrals	711	5585	Benzo(b)fluoranthene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5601	Benzo(b+k)fluoranthene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass		
WP Base/Neutrals	711	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5600	Benzo(k)fluoranthene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5600	Benzo(k)fluoranthene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5600	Benzo(k)fluoranthene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5600	Benzo(k)fluoranthene	EPA 8270C	Pass		
WP Base/Neutrals	711	5600	Benzo(k)fluoranthene	EPA 8270C	Pass		
Acid Extractables	4190	5610	Benzoic Acid	EPA 8270C	Pass		
Acids	712	5610	Benzoic acid	EPA 8270C	Pass		
Base Neutral Extractables	4200	5630	Benzyl alcohol	EPA 8270C	Pass		
WP Base/Neutrals	711	5630	Benzyl alcohol	EPA 8270C	Pass		
Base Neutral Extractables	4200	5670	Benzyl butyl phthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	5760	bis(2-Chloroethoxy) methane	EPA 8270C	Pass		
WP Base/Neutrals	711	5760	bis(2-Chloroethoxy) methane	EPA 8270C	Pass		
Base Neutral Extractables	4200	5765	bis(2-Chloroethyl) ether	EPA 8270C	Pass		
WP Base/Neutrals	711	5765	bis(2-Chloroethyl) ether	EPA 8270C	Pass		
Base Neutrals Extractables	4200	5780	bis(2-Chloroisopropyl) ether	EPA 8270C	Pass		
WP Base/Neutrals	711	5780	bis(2-Chloroisopropyl) ether	EPA 8270C	Pass		
Base Neutral Extractables	4200	6255	bis(2-Ethylhexyl) phthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	6255	bis(2-Ethylhexyl) phthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	5670	Butylbenzylphthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	5680	Carbazole	EPA 8270C	Pass		
WP Base/Neutrals	711	5680	Carbazole	EPA 8270C	Pass		
Base Neutral Extractables	4200	5855	Chrysene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5855	Chrysene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5855	Chrysene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5855	Chrysene	EPA 8270C	Pass		
WP Base/Neutrals	711	5855	Chrysene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass		
WP Base/Neutrals	711	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5905	Dibenzofuran	EPA 8270C	Pass		
WP Base/Neutrals	711	5905	Dibenzofuran	EPA 8270C	Pass		
Base Neutral Extractables	4200	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6070	Diethyl phthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	6070	Diethylphthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	6135	Dimethyl phthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	6135	Dimethyl phthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	5925	Di-n-butylphthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	5925	Di-n-butylphthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	6200	Di-n-octylphthalate	EPA 8270C	Pass		
WP Base/Neutrals	711	6200	Di-n-octylphthalate	EPA 8270C	Pass		
Base Neutral Extractables	4200	6265	Fluoranthene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	6265	Fluoranthene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	6265	Fluoranthene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	6265	Fluoranthene	EPA 8270C	Pass		
WP Base/Neutrals	711	6265	Fluoranthene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6270	Fluorene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	6270	Fluorene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	6270	Fluorene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	6270	Fluorene	EPA 8270C	Pass		
WP Base/Neutrals	711	6270	Fluorene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6275	Hexachlorobenzene	EPA 8270C	Pass		
WP Base/Neutrals	711	6275	Hexachlorobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	4835	Hexachlorobutadiene	EPA 8270C	Pass		
WP Base/Neutrals	711	4835	Hexachlorobutadiene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6285	Hexachlorocyclopentadiene	EPA 8270C	Pass		

WP Base/Neutrals	711	6285	Hexachlorocyclopentadiene	EPA 8270C	Pass		
Base Neutral Extractables	4200	4840	Hexachloroethane	EPA 8270C	Pass		
WP Base/Neutrals	711	4840	Hexachloroethane	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass		
Base Neutral Extractables	4200	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass		
WP Base/Neutrals	711	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass		
WP Base/Neutrals	711	6320	Isophorone	EPA 8270C	Pass		
Base Neutral Extractables	4200	6320	Isophorone	EPA 8270C	Pass		
Base Neutral Extractables	4200	5005	Naphthalene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	5005	Naphthalene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	5005	Naphthalene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	5005	Naphthalene	EPA 8270C	Pass		
WP Base/Neutrals	711	5005	Naphthalene	EPA 8270C	Pass		
WP Base/Neutrals	711	5015	Nitrobenzene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5015	Nitrobenzene (NB)	EPA 8270C	Pass		
Base Neutral Extractables	4200	6530	N-nitrosodimethylamine	EPA 8270C	Pass		
WP Base/Neutrals	711	6530	N-Nitrosodimethylamine	EPA 8270C	Pass		
Base Neutral Extractables	4200	6545	N-Nitroso-di-n-propylamine	EPA 8270C	Pass		
WP Base/Neutrals	711	6545	N-Nitroso-di-n-propylamine	EPA 8270C	Pass		
Base Neutral Extractables	4200	6535	N-nitrosodiphenylamine	EPA 8270C	Pass		
WP Base/Neutrals	711	6535	N-Nitrosodiphenylamine	EPA 8270C	Pass		
Acid Extractables	4190	6605	Pentachlorophenol	EPA 8270C	Pass		
Acids	712	6605	Pentachlorophenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	6615	Phenanthrene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	6615	Phenanthrene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	6615	Phenanthrene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	6615	Phenanthrene	EPA 8270C	Pass		
WP Base/Neutrals	711	6615	Phenanthrene	EPA 8270C	Pass		
Acid Extractables	4190	6625	Phenol	EPA 8270C	Pass		
Acids	712	6625	Phenol	EPA 8270C	Pass		
Base Neutral Extractables	4200	6665	Pyrene	EPA 8270C	Pass		
Base/Neutrals 1	PEO-121-1	6665	Pyrene	EPA 8270C	Pass		
CWA Low Level PAH Mix	38010	6665	Pyrene	EPA 8270C	Pass		
PAH-GC & GCMS	4880	6665	Pyrene	EPA 8270C	Pass		
WP Base/Neutrals	711	6665	Pyrene	EPA 8270C	Pass		
Base Neutral Extractables	4200	5095	Pyridine	EPA 8270C	Pass		
WP Base/Neutrals	711	5095	Pyridine	EPA 8270C	Pass		
Base Neutral Extractables	4200	6715	1,2,4,5-Tetrachlorobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5155	1,2,4-Trichlorobenzene	EPA 8270D	Pass		
WP Base/Neutrals	711	5155	1,2,4-Trichlorobenzene	EPA 8270D	Pass		
WP Base/Neutrals	711	4610	1,2-Dichlorobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	4610	1,2-Dichlorobenzene	EPA 8270D	Pass		
WP Base/Neutrals	711	4615	1,3-Dichlorobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	4615	1,3-Dichlorobenzene	EPA 8270D	Pass		
WP Base/Neutrals	711	4620	1,4-Dichlorobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	4620	1,4-Dichlorobenzene	EPA 8270D	Pass		
Acids	712	6735	2,3,4,6-Tetrachlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6735	2,3,4,6-Tetrachlorophenol	EPA 8270D	Pass		
Acids	712	6835	2,4,5-Trichlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6835	2,4,5-Trichlorophenol	EPA 8270D	Pass		
Acids	712	6840	2,4,6-Trichlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6840	2,4,6-Trichlorophenol	EPA 8270D	Pass		
Acids	712	6000	2,4-Dichlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6000	2,4-Dichlorophenol	EPA 8270D	Pass		
Acids	712	6130	2,4-Dimethylphenol	EPA 8270D	Pass		
Acid Extractables	4190	6130	2,4-Dimethylphenol	EPA 8270D	Pass		
Acids	712	6175	2,4-Dinitrophenol	EPA 8270D	Pass		
Acid Extractables	4190	6175	2,4-Dinitrophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6185	2,4-Dinitrotoluene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 8270D	Pass		
Acids	712	6005	2,6-Dichlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6005	2,6-Dichlorophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6190	2,6-Dinitrotoluene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6190	2,6-Dinitrotoluene (2,6-DNT)	EPA 8270D	Pass		
WP Base/Neutrals	711	5795	2-Chloronaphthalene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5795	2-Chloronaphthalene	EPA 8270D	Pass		
Acids	712	5800	2-Chlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	5800	2-Chlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6360	2-Methyl-4,6-Dinitrophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6385	2-Methylnaphthalene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6385	2-Methylnaphthalene	EPA 8270D	Pass		
Acids	712	6400	2-Methylphenol	EPA 8270D	Pass		
Acid Extractables	4190	6400	2-Methylphenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6460	2-Nitroaniline	EPA 8270D	Pass		
Base Neutral Extractables	4200	6460	2-Nitroaniline	EPA 8270D	Pass		
Acids	712	6490	2-Nitrophenol	EPA 8270D	Pass		
Acid Extractables	4190	6490	2-Nitrophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	5945	3,3'-Dichlorobenzidine	EPA 8270D	Pass		
Base Neutral Extractables	4200	5945	3,3'-Dichlorobenzidine	EPA 8270D	Pass		
Acid Extractables	4190	6405	3-Methylphenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6465	3-Nitroaniline	EPA 8270D	Pass		
Base Neutral Extractables	4200	6465	3-Nitroaniline	EPA 8270D	Pass		
Acids	712	6360	4,6-Dinitro-2-methylphenol	EPA 8270D	Pass		
Base Neutral Extractables	4200	5660	4-Bromophenyl phenyl ether	EPA 8270D	Pass		
WP Base/Neutrals	711	5660	4-Bromophenyl-phenylether	EPA 8270D	Pass		
Acids	712	5700	4-Chloro-3-methylphenol	EPA 8270D	Pass		
Acid Extractables	4190	5700	4-Chloro-3-methylphenol	EPA 8270D	Pass		
WP Base/Neutrals	711	5745	4-Chloroaniline	EPA 8270D	Pass		
Base Neutral Extractables	4200	5745	4-Chloroaniline	EPA 8270D	Pass		

WP Base/Neutrals	711	5825	4-Chlorophenyl-phenylether	EPA 8270D	Pass		
Base Neutral Extractables	4200	5825	4-Chlorophenyl-phenylether	EPA 8270D	Pass		
Acids	712	6410	4-Methylphenol	EPA 8270D	Pass		
Acid Extractables	4190	6410	4-Methylphenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6470	4-Nitroaniline	EPA 8270D	Pass		
Base Neutral Extractables	4200	6470	4-Nitroaniline	EPA 8270D	Pass		
Acids	712	6500	4-Nitrophenol	EPA 8270D	Pass		
Acid Extractables	4190	6500	4-Nitrophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	5500	Acenaphthene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5500	Acenaphthene	EPA 8270D	Pass		
WP Base/Neutrals	711	5505	Acenaphthylene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5505	Acenaphthylene	EPA 8270D	Pass		
WP Base/Neutrals	711	5545	Aniline	EPA 8270D	Pass		
Base Neutral Extractables	4200	5545	Aniline	EPA 8270D	Pass		
WP Base/Neutrals	711	5555	Anthracene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5555	Anthracene	EPA 8270D	Pass		
WP Base/Neutrals	711	5595	Benzidine	EPA 8270D	Pass		
Base Neutral Extractables	4200	5595	Benzidine	EPA 8270D	Pass		
WP Base/Neutrals	711	5575	Benzo(a)anthracene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5575	Benzo(a)anthracene	EPA 8270D	Pass		
WP Base/Neutrals	711	5580	Benzo(a)pyrene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5580	Benzo(a)pyrene	EPA 8270D	Pass		
WP Base/Neutrals	711	5585	Benzo(b)fluoranthene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5585	Benzo(b)fluoranthene	EPA 8270D	Pass		
WP Base/Neutrals	711	5590	Benzo(g,h,l)perylene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5590	Benzo(g,h,l)perylene	EPA 8270D	Pass		
WP Base/Neutrals	711	5600	Benzo(k)fluoranthene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5600	Benzo(k)fluoranthene	EPA 8270D	Pass		
Acids	712	5610	Benzoic acid	EPA 8270D	Pass		
Acid Extractables	4190	5610	Benzoic Acid	EPA 8270D	Pass		
WP Base/Neutrals	711	5630	Benzyl alcohol	EPA 8270D	Pass		
Base Neutral Extractables	4200	5630	Benzyl alcohol	EPA 8270D	Pass		
Base Neutral Extractables	4200	5670	Benzyl butyl phthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	5760	bis(2-Chloroethoxy) methane	EPA 8270D	Pass		
Base Neutral Extractables	4200	5760	bis(2-Chloroethoxy) methane	EPA 8270D	Pass		
Base Neutral Extractables	4200	5765	bis(2-Chloroethyl) ether	EPA 8270D	Pass		
WP Base/Neutrals	711	5765	bis(2-Chloroethyl)ether	EPA 8270D	Pass		
WP Base/Neutrals	711	5780	bis(2-Chloroisopropyl) ether	EPA 8270D	Pass		
Base Neutral Extractables	4200	5780	bis(2-Chloroisopropyl) ether	EPA 8270D	Pass		
WP Base/Neutrals	711	6255	bis(2-Ethylhexyl) phthalate	EPA 8270D	Pass		
Base Neutral Extractables	4200	6255	bis(2-Ethylhexyl) phthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	5670	Butylbenzylphthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	5680	Carbazole	EPA 8270D	Pass		
Base Neutral Extractables	4200	5680	Carbazole	EPA 8270D	Pass		
WP Base/Neutrals	711	5855	Chrysene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5855	Chrysene	EPA 8270D	Pass		
WP Base/Neutrals	711	5895	Dibenz(a,h) anthracene	EPA 8270D	Pass		
WP Base/Neutrals	711	5905	Dibenzofuran	EPA 8270D	Pass		
Base Neutral Extractables	4200	5905	Dibenzofuran	EPA 8270D	Pass		
Base Neutral Extractables	4200	5895	Dibenz(a,h) anthracene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6070	Diethyl phthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	6070	Diethylphthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	6135	Dimethyl phthalate	EPA 8270D	Pass		
Base Neutral Extractables	4200	6135	Dimethyl phthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	5925	Di-n-butylphthalate	EPA 8270D	Pass		
Base Neutral Extractables	4200	5925	Di-n-butylphthalate	EPA 8270D	Pass		
WP Base/Neutrals	711	6200	Di-n-octylphthalate	EPA 8270D	Pass		
Base Neutral Extractables	4200	6200	Di-n-octylphthalate	EPA 8270D	Pass		
Base Neutral Extractables	4200	6265	Fluoranthene	EPA 8270D	Pass		
WP Base/Neutrals	711	6265	Fluoranthene	EPA 8270D	Pass		
WP Base/Neutrals	711	6270	Fluorene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6270	Fluorene	EPA 8270D	Pass		
WP Base/Neutrals	711	6275	Hexachlorobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6275	Hexachlorobenzene	EPA 8270D	Pass		
WP Base/Neutrals	711	4835	Hexachlorobutadiene	EPA 8270D	Pass		
Base Neutral Extractables	4200	4835	Hexachlorobutadiene	EPA 8270D	Pass		
WP Base/Neutrals	711	6285	Hexachlorocyclopentadiene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6285	Hexachlorocyclopentadiene	EPA 8270D	Pass		
Base Neutral Extractables	4200	4840	Hexachloroethane	EPA 8270D	Pass		
WP Base/Neutrals	711	4840	Hexachloroethane	EPA 8270D	Pass		
WP Base/Neutrals	711	6315	Indeno (1,2,3-cd) pyrene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6315	Indeno (1,2,3-cd) pyrene	EPA 8270D	Pass		
WP Base/Neutrals	711	6320	Isophorone	EPA 8270D	Pass		
Base Neutral Extractables	4200	6320	Isophorone	EPA 8270D	Pass		
WP Base/Neutrals	711	5005	Naphthalene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5005	Naphthalene	EPA 8270D	Pass		
WP Base/Neutrals	711	5015	Nitrobenzene	EPA 8270D	Pass		
Base Neutral Extractables	4200	5015	Nitrobenzene (NB)	EPA 8270D	Pass		
WP Base/Neutrals	711	6530	N-Nitrosodimethylamine	EPA 8270D	Pass		
Base Neutral Extractables	4200	6530	N-nitrosodimethylamine	EPA 8270D	Pass		
WP Base/Neutrals	711	6545	N-Nitroso-di-n-propylamine	EPA 8270D	Pass		
Base Neutral Extractables	4200	6545	N-Nitroso-di-n-propylamine	EPA 8270D	Pass		
WP Base/Neutrals	711	6535	N-Nitrosodiphenylamine	EPA 8270D	Pass		
Base Neutral Extractables	4200	6535	N-nitrosodiphenylamine	EPA 8270D	Pass		
Acids	712	6605	Pentachlorophenol	EPA 8270D	Pass		
Acid Extractables	4190	6605	Pentachlorophenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6615	Phenanthrene	EPA 8270D	Pass		
Base Neutral Extractables	4200	6615	Phenanthrene	EPA 8270D	Pass		
Acids	712	6625	Phenol	EPA 8270D	Pass		
Acid Extractables	4190	6625	Phenol	EPA 8270D	Pass		
WP Base/Neutrals	711	6665	Pyrene	EPA 8270D	Pass		

Base Neutral Extractables	4200	6665	Pyrene	EPA 8270D	Pass		
WP Base/Neutrals	711	5095	Pyridine	EPA 8270D	Pass		
Base Neutral Extractables	4200	5095	Pyridine	EPA 8270D	Pass		
Dioxin	PEO-258	9420	1,2,3,4,6,7,8-HpCDF	EPA 8290	Pass		
2,3,7,8-Tetrachlorodibenzo-p-dioxin	38186	9618	2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290	Pass		
WP Carbamates	38156	7205	Carbofuran	EPA 8321A	Pass		
WP Carbamates	38156	7505	Diuron	EPA 8321A	Pass		
WP Carbamates	38156	7750	Methomyl	EPA 8321A	Pass		
WP Carbamates	38156	7940	Oxamyl	EPA 8321A	Pass		
WP Carbamates	38156	8075	Propham	EPA 8321A	Pass		
Herbicides	PEO-094	8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8321A	Pass		
CWA Nitroaromatics in Water	38172	6885	1,3,5-Trinitrobenzene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	6160	1,3-Dinitrobenzene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9651	2,4,6-Trinitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	6185	2,4-Dinitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	6190	2,6-Dinitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9303	2-Amino-4,6-dinitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9507	2-Nitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9510	3-Nitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9306	4-Amino-2,6-dinitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9513	4-Nitrotoluene	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9522	HMX	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	9432	RDX	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	6415	Tetryl	EPA 8330	Pass		
CWA Nitroaromatics in Water	38172	6885	1,3,5-Trinitrobenzene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6885	1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	6160	1,3-Dinitrobenzene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6160	1,3-Dinitrobenzene (1,3-DNB)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9651	2,4,6-Trinitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9651	2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	6190	2,6-Dinitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6190	2,6-Dinitrotoluene (2,6-DNT)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9303	2-Amino-4,6-dinitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9303	2-Amino-4,6-dinitrotoluene (2am-dnt)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9507	2-Nitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9507	2-Nitrotoluene	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9510	3-Nitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9510	3-Nitrotoluene	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9306	4-Amino-2,6-dinitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9306	4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9513	4-Nitrotoluene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9513	4-Nitrotoluene	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9522	HMX	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9522	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	5015	Nitrobenzene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	5015	Nitrobenzene	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6485	Nitroglycerin	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	9432	RDX	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	9432	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330A	Pass		
CWA Nitroaromatics in Water	38172	6415	Tetryl	EPA 8330A	Pass		
Low Level Nit/Nit	PEO-251	6415	Tetryl (Methyl-2,4,6-trinitrophenylnitramine)	EPA 8330A	Pass		
Total Cyanide	4090	1645	Total Cyanide	EPA 9010B	Pass		
Total Cyanide	PEI-031	1645	Total cyanide	EPA 9010B	Pass		
Total Cyanide	4090	1645	Total Cyanide	EPA 9014	Pass		
CWA Anions	55131	1540	Bromide (Br)	EPA 9056	Pass		
WP Minerals #1	55144	1575	Chloride	EPA 9056	Pass		
Fluoride	4420	1730	Fluoride	EPA 9056	Pass		
WP Minerals #2	55145	1730	Fluoride	EPA 9056	Pass		
WP & DMRQA Nutrients	55035	1810	Nitrate as N	EPA 9056	Pass		
Nutrients	4020	1810	Nitrate Nitrogen as N	EPA 9056	Pass		
Nitrate-Nitrite as N	4770	1820	Nitrate-Nitrite as N	EPA 9056	Pass		
Nitrite as N	4780	1840	Nitrite as N	EPA 9056	Pass		
Nutrients	4020	1870	Orthophosphate as P	EPA 9056	Pass		
WP & DMRQA Nutrients	55035	1870	Orthophosphate as P	EPA 9056	Pass		
Miscellaneous Analytes	PEI-051	1540	Bromide	EPA 9056	Pass		
Minerals	PEI-051	1575	Chloride	EPA 9056	Pass		
Minerals	PEI-051	1730	Fluoride	EPA 9056	Pass		
Nutrients	PEI-051	1805	Nitrate as N	EPA 9056	Pass		
Nutrients	PEI-051	1820	Nitrate+nitrite as N	EPA 9056	Pass		
Nutrients	PEI-051	1840	Nitrite as N	EPA 9056	Pass		
Nutrients	PEI-051	1870	Orthophosphate as P	EPA 9056	Pass		
Minerals	PEI-051	2000	Sulfate	EPA 9056	Pass		
Demand	4010	2040	Total organic carbon (TOC)	EPA 9060	Pass		
Demands	PEI-026	2040	Total organic carbon (TOC)	EPA 9060	Pass		
Fluoride	4420	1730	Fluoride	EPA 9214	Pass		
Minerals	4050	1505	Alkalinity as CaCO3	SM 2320B	Pass		
Solids (Total Solids, TSS, & TDS)	55085	1955	Total Dissolved Solids (TDS)	SM 2540C	Pass		
Solids	4030	1705	Total Dissolved Solids at 180C	SM 2540C	Pass		
Solids	4030	1960	Total Suspended Solids	SM 2540D	Pass		
CWA UV 254 Absorbance/ DOC	55088	1710	Dissolved Organic Carbon	SM 5310B	Pass		
Demand	4010	2040	Total Organic Carbon	SM 5310B	Pass		
WP & DMRQA Demands	55055	2040	Total Organic Carbon	SM 5310B	Pass		
Demands	PEI-026	2040	Total organic carbon (TOC)	SM 5310B	Pass		
Oil & Grease	4120	1860	Oil & Grease	SM 5520B	Pass		
MBAS	4430	2025	MBAS	SM 5540C	Pass		
WP MBAS	55083	2025	MBAS	SM 5540C	Pass		

DoD ELAP -- PT Performance Summary Review -- WS ALL

PartName	PartNumber	NELACCCode	AnalyteName	EPAmethod#	PT results - Pass/Acceptable Results
Lab Name :	APPL, Inc.				
City/State :	Clovis, CA				
PT Provider Used :	ERA, Absolute, RTC, APG				
WS Chromium VI	55112	1045	Chromium VI	EPA 218.6	Pass
Trace Metals	5070	1095	Mercury	EPA 245.1	Pass
WS Trace Elements	55012	1095	Total Mercury	EPA 245.1	Pass
WS Inorganic Disinfection By-Products	55010	1540	Bromide	EPA 300.0	Pass
Minerals	5080	1575	Chloride	EPA 300.0	Pass
WS Minerals Mix #1	55122	1575	Chloride	EPA 300.0	Pass
Nutrients	5140	1730	Fluoride	EPA 300.0	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1730	Fluoride	EPA 300.0	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1820	Nitrate and Nitrite as N	EPA 300.0	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1810	Nitrate as N	EPA 300.0	Pass
Nutrients	5140	1810	Nitrate Nitrogen as N	EPA 300.0	Pass
Nitrate+Nitrite as N	5860	1820	Nitrate+Nitrite as N	EPA 300.0	Pass
Nutrients	5140	1840	Nitrite as N	EPA 300.0	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1840	Nitrite as N	EPA 300.0	Pass
Nutrients	5140	1870	Orthophosphate as P	EPA 300.0	Pass
Minerals	5080	2000	Sulfate	EPA 300.0	Pass
WS Sulphate/TOC	55070	2000	Sulfate	EPA 300.0	Pass
Perchlorate	5610	1895	Perchlorate	EPA 314.0	Pass
WS Perchlorate	55099	1895	Perchlorate	EPA 314.0	Pass
Nutrients	5140	1730	Fluoride	EPA 340.2	Pass
SDWA Nutrients	55165	1515	Ammonia as N	EPA 350.1	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1820	Nitrate and Nitrite as N	EPA 353.2	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1810	Nitrate as N	EPA 353.2	Pass
Nutrients	5140	1810	Nitrate Nitrogen as N	EPA 353.2	Pass
Nitrate+Nitrite as N	5860	1820	Nitrate+Nitrite as N	EPA 353.2	Pass
Nutrients	5140	1840	Nitrite as N	EPA 353.2	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1840	Nitrite as N	EPA 353.2	Pass
WS NO3-, NO2-, F, PO4-3, and NO3- & NO2- as N	55011	1870	Orthophosphate as P	EPA 353.2	Pass
Nutrients	5140	1870	Orthophosphate as P	EPA 353.2	Pass
SDWA Nutrients	55165	1910	Total Phosphorus	EPA 365.2	Pass
MBAS	5470	2025	MBAS	EPA 425.1	Pass
Perchlorate	5610	1895	Perchlorate	EPA 6850	Pass
WS Perchlorate	55099	1895	Perchlorate	EPA 6850	Pass
pH	5060	1900	pH	EPA 9040B	Pass
UV 254/DOC	5480	1710	Dissolved Organic Carbon (DOC)	EPA 9060	Pass
Total Organic Carbon (TOC)	5250	2040	Total Organic Carbon	EPA 9060	Pass
Nutrients	5140	1730	Fluoride	EPA 9214	Pass
Minerals	5080	1505	Alkalinity	SM 2320B	Pass
Minerals	5080	1955	Total Dissolved Solids	SM 2540C	Pass
SDWA Solids (Total Solids, TSS, & TDS)	55161	1955	Total Dissolved Solids	SM 2540C	Pass
Solids	5150	1705	Total Dissolved Solids	SM 2540C	Pass
SDWA Solids (Total Solids, TSS, & TDS)	55161	1960	Non-Filterable Residue (TSS)	SM 2540D	Pass
Solids	5150	1960	Total Suspended Solids	SM 2540D	Pass
MBAS	5470	2025	MBAS	SM 5540C	Pass
WS MBAS	55106	2025	MBAS	SM 5540C	Pass

DoD ELAP -- PT Performance Summary Review -- SOIL						
Lab Name :		APPL, Inc.				
City/State :		Clovis, CA				
PT Provider Used :		ERA, RTC, Absolute, APG				
PartName	PartNumber	NELACCode	AnalyteName	EPAMethod#	PT results - Pass/Acceptable Results	
Oil & Grease - n-Hexadecane & Stearic acid	55084	1860	Oil & Grease	EPA 1664A	Pass	
PCB Congeners in Soil	SPE-068	9070	2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9025	2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9040	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8980	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8955	2,2',5,5'-Tetrachlorobiphenyl (PCB 52)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9085	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9050	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 156)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9045	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8985	2,3,3',4,4',5'-Pentachlorobiphenyl (PCB 105)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9055	2,3',4,4',5,5'-Hexachlorobiphenyl (PCB 167)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9005	2,3,4,4',5'-Pentachlorobiphenyl (PCB 114)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8995	2,3',4,4',5'-Pentachlorobiphenyl (PCB 118)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9000	2,3',4,4',5'-Pentachlorobiphenyl (PCB 123)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8936	2,4,4'-Trichlorobiphenyl (PCB 28)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9060	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9015	3,3',4,4',5'-Pentachlorobiphenyl (PCB 126)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8965	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8970	3,4,4',5'-Tetrachlorobiphenyl (PCB 81)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9025	PCB (129)+(138)+(163)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9040	PCB (153)+(168)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9046	PCB (156)+(157)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	9070	PCB (180)+(193)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8936	PCB (20)+(28)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8980	PCB (90)+(101)+(113)	EPA 1668	Pass	
PCB Congeners in Soil	SPE-068	8870	PCBs, total	EPA 1668	Pass	
RCRA Anions	55141	1540	Bromide	EPA 300.0	Pass	
RCRA Anions	55141	1575	Chloride	EPA 300.0	Pass	
RCRA Anions	55141	1730	Fluoride	EPA 300.0	Pass	
RCRA Anions	55141	1810	Nitrate as N	EPA 300.0	Pass	
RCRA Anions	55141	1870	Phosphate as P	EPA 300.0	Pass	
RCRA Anions	55141	2000	Sulfate	EPA 300.0	Pass	
RCRA Hexavalent Chromium	55104	1045	Chromium IV	EPA 3060A	Pass	
Hexavalent Chromium in Soil	4120	1045	Chromium, Hexavalent	EPA 3060A	Pass	
RCRA Perchlorate	38151	1885	Perchlorate	EPA 314.0	Pass	
RCRA Nutrients	55142	1515	Ammonia as N	EPA 350.1	Pass	
Nutrients in Soil	4170	1515	Ammonia Nitrogen as N	EPA 350.1	Pass	
Nutrients in Soil	4170	1795	Total Kjeldahl Nitrogen	EPA 351.2	Pass	
RCRA Nutrients	55142	1795	Total Kjeldahl Nitrogen	EPA 351.2	Pass	
RCRA Metals In Soil #2	55103	1000	Aluminum	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1005	Antimony	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1005	Antimony	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1005	Antimony, Sb	EPA 6010B	Pass	
TCLP Metals in Soil - CA WET	SPE-006	1005	Antimony, Sb	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1010	Arsenic	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1010	Arsenic, As	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1015	Barium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1015	Barium	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1015	Barium, Ba	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1020	Beryllium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1020	Beryllium	EPA 6010B	Pass	
TCLP Metals in Soil - CA WET	SPE-006	1020	Beryllium, Be	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1025	Boron	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1030	Cadmium	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1030	Cadmium, Cd	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1030	Cadmium	EPA 6010B	Pass	
RCRA Metals in Soil #2	55103	1035	Calcium	EPA 6010B	Pass	
RCRA Metals In Soil #2	55103	1035	Calcium	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1040	Chromium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1040	Chromium	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1040	Chromium, Cr (total)	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1050	Cobalt	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1055	Copper	EPA 6010B	Pass	
TCLP Metals in Soil - CA WET	SPE-006	1055	Copper, Cu	EPA 6010B	Pass	
RCRA Metals In Soil #2	55103	1070	Iron	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1075	Lead	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1075	Lead	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1075	Lead, Pb	EPA 6010B	Pass	
RCRA Metals In Soil #2	55103	1085	Magnesium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1090	Manganese	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1100	Molybdenum	EPA 6010B	Pass	
TCLP Metals in Soil - CA WET	SPE-006	1100	Molybdenum, Mo	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1105	Nickel	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1105	Nickel	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1105	Nickel, Ni	EPA 6010B	Pass	
RCRA Metals In Soil #2	55103	1125	Potassium	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1140	Selenium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1140	Selenium	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1140	Selenium, Se	EPA 6010B	Pass	
TCLP Metals	PT-TCLPMET-SOIL	1150	Silver	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1150	Silver	EPA 6010B	Pass	
TCLP Metals in Soil	4180	1150	Silver, Ag	EPA 6010B	Pass	
RCRA Metals In Soil #2	55103	1155	Sodium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1160	Strontium	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1165	Thallium	EPA 6010B	Pass	
TCLP Metals in Soil - CA WET	SPE-006	1165	Thallium, Tl	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1175	Tin	EPA 6010B	Pass	
RCRA Metals in Soil #1	55102	1180	Titanium	EPA 6010B	Pass	
RCRA Nutrients	55142	1910	Total Phosphorus	EPA 6010B	Pass	
Nutrients in Soil	4170	1910	Total Phosphorus as P	EPA 6010B	Pass	

RCRA Metals in Soil #1	55102	1185	Vanadium	EPA 6010B	Pass
TCLP Metals in Soil - CA WET	SPE-006	1185	Vanadium, V	EPA 6010B	Pass
TCLP Metals	PT-TCLPMET-SOIL	1190	Zinc	EPA 6010B	Pass
RCRA Metals in Soil #1	55102	1190	Zinc	EPA 6010B	Pass
TCLP Metals in Soil	4180	1190	Zinc, Zn	EPA 6010B	Pass
RCRA Metals in Soil #2	55103	1000	Aluminum	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1005	Antimony	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1015	Barium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1020	Beryllium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1025	Boron	EPA 6020	Pass
RCRA Metals in Soil #2	55103	1035	Calcium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1040	Chromium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1050	Cobalt	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1055	Copper	EPA 6020	Pass
RCRA Metals in Soil #2	55103	1070	Iron	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1075	Lead	EPA 6020	Pass
RCRA Metals in Soil #2	55103	1085	Magnesium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1090	Manganese	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1100	Molybdenum	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1105	Nickel	EPA 6020	Pass
RCRA Metals in Soil #2	55103	1125	Potassium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1140	Selenium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1150	Silver	EPA 6020	Pass
RCRA Metals in Soil #2	55103	1155	Sodium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1160	Strontium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1165	Thallium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1175	Tin	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1180	Titanium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1185	Vanadium	EPA 6020	Pass
RCRA Metals in Soil #1	55102	1190	Zinc	EPA 6020	Pass
RCRA Perchlorate	55143	1895	Perchlorate	EPA 6850	Pass
RCRA Hexavalent Chromium	55104	1045	Chromium VI	EPA 7196A	Pass
Hexavalent Chromium in Soil	4120	1045	Chromium, Hexavalent	EPA 7196A	Pass
RCRA Hexavalent Chromium	55104	1045	Chromium VI	EPA 7199	Pass
Hexavalent Chromium in Soil	4120	1045	Chromium, Hexavalent	EPA 7199	Pass
TCLP Metals	PT-TCLPMET-SOIL	1095	Mercury	EPA 7470A	Pass
TCLP Metals in Soil	4180	1095	Mercury	EPA 7470A	Pass
RCRA Metals in Soil #1	55102	1095	Mercury	EPA 7471A	Pass
RCRA Metals in Soil #1	55102	1095	Mercury	EPA 7471B	Pass
Diesel Fuel #2 in Soil	38115	9369	#2 Fuel Oil (Diesel)	EPA 8015B	Pass
PT Diesel Fuel #2 in Water	38114	9369	#2 Fuel Oil (Diesel)	EPA 8015B	Pass
93 Octane Gasoline in Soil	38117	9408	93 Octane Gasoline in Soil	EPA 8015B	Pass
PT Unleaded Gasoline in Water	38116	9408	Unleaded Gasoline 93 Octane	EPA 8015B	Pass
RCRA BTEX & MTBE	38161	4375	Benzene	EPA 8021B	Pass
RCRA BTEX & MTBE	38161	4765	Ethyl benzene	EPA 8021B	Pass
RCRA BTEX & MTBE	38161	5000	Methyl tert-butyl ether (MTBE)	EPA 8021B	Pass
RCRA BTEX & MTBE	38161	5140	Toluene	EPA 8021B	Pass
RCRA BTEX & MTBE	38161	5260	Total Xylenes	EPA 8021B	Pass
Chlorinated Pesticides in Soil	38101	7355	4,4'-DDD	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7360	4,4'-DDE	EPA 8081A	Pass
Pesticides in Soil	14221	7360	4,4'-DDE	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7365	4,4'-DDT	EPA 8081A	Pass
Pesticides in Soil	14222	7365	4,4'-DDT	EPA 8081A	Pass
Pesticides in Soil	14220	7355	4,4'-DDD	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7110	a-BHC	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7240	a-Chlordane	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7025	Aldrin	EPA 8081A	Pass
Pesticides in Soil	14223	7025	Aldrin	EPA 8081A	Pass
Pesticides in Soil	14224	7110	alpha-BHC	EPA 8081A	Pass
Pesticides in Soil	14225	7240	alpha-Chlordane	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7115	b-BHC	EPA 8081A	Pass
Pesticides in Soil	14226	7115	beta-BHC	EPA 8081A	Pass
Chlordane in Soil	38141	7250	Chlordane	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7105	d-BHC	EPA 8081A	Pass
Pesticides in Soil	14227	7105	delta-BHC	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7470	Dieldrin	EPA 8081A	Pass
Pesticides in Soil	14228	7470	Dieldrin	EPA 8081A	Pass
Pesticides in Soil	14229	7510	Endosulfan I	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7515	Endosulfan II	EPA 8081A	Pass
Pesticides in Soil	14230	7515	Endosulfan II	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7520	Endosulfan sulfate	EPA 8081A	Pass
Pesticides in Soil	14231	7520	Endosulfan sulfate	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7540	Endrin	EPA 8081A	Pass
Pesticides in Soil	14232	7540	Endrin	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7530	Endrin aldehyde	EPA 8081A	Pass
Pesticides in Soil	14233	7530	Endrin aldehyde	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7535	Endrin ketone	EPA 8081A	Pass
Pesticides in Soil	14234	7535	Endrin Ketone	EPA 8081A	Pass
Pesticides in Soil	14235	7120	gamma-BHC (Lindane)	EPA 8081A	Pass
Pesticides in Soil	14236	7245	gamma-Chlordane	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7120	g-BHC (Lindane)	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7685	Heptachlor	EPA 8081A	Pass
Pesticides in Soil	14237	7685	Heptachlor	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7690	Heptachlor epoxide	EPA 8081A	Pass
Pesticides in Soil	14238	7690	Heptachlor epoxide	EPA 8081A	Pass
Chlorinated Pesticides in Soil	38101	7810	Methoxychlor	EPA 8081A	Pass
Pesticides in Soil	14239	7810	Methoxychlor	EPA 8081A	Pass
Total Chlordane in Soil	4240	7250	Total Chlordane	EPA 8081A	Pass
Toxaphene in Soil	38066	8250	Toxaphene	EPA 8081A	Pass
Toxaphene in Soil	4230	8250	Toxaphene	EPA 8081A	Pass
Aroclor in Soil	38142	8880	Aroclor 1016	EPA 8082	Pass
WP PCBs in Water	38091	8880	Aroclor 1016	EPA 8082	Pass
WP PCBs in Water	38094	8880	Aroclor 1016	EPA 8082	Pass
Aroclor in Soil	38142	8885	Aroclor 1221	EPA 8082	Pass
WP PCBs in Water	38091	8885	Aroclor 1221	EPA 8082	Pass
WP PCBs in Water	38094	8885	Aroclor 1221	EPA 8082	Pass
Aroclor in Soil	38142	8890	Aroclor 1232	EPA 8082	Pass
WP PCBs in Water	38091	8890	Aroclor 1232	EPA 8082	Pass

WP PCBs in Water	38094	8890	Aroclor 1232	EPA 8082	Pass
Aroclor in Soil	38142	8895	Aroclor 1242	EPA 8082	Pass
WP PCBs in Water	38091	8895	Aroclor 1242	EPA 8082	Pass
WP PCBs in Water	38094	8895	Aroclor 1242	EPA 8082	Pass
Aroclor in Soil	38142	8900	Aroclor 1248	EPA 8082	Pass
WP PCBs in Water	38091	8900	Aroclor 1248	EPA 8082	Pass
WP PCBs in Water	38094	8900	Aroclor 1248	EPA 8082	Pass
Aroclor in Soil	38142	8905	Aroclor 1254	EPA 8082	Pass
WP PCBs in Water	38091	8905	Aroclor 1254	EPA 8082	Pass
WP PCBs in Water	38094	8905	Aroclor 1254	EPA 8082	Pass
Aroclor in Soil	38142	8910	Aroclor 1260	EPA 8082	Pass
WP PCBs in Water	38091	8910	Aroclor 1260	EPA 8082	Pass
WP PCBs in Water	38094	8910	Aroclor 1260	EPA 8082	Pass
PCB in Soil	SPE-010	8912	Aroclor 1016/1242	EPA 8082	Pass
PCB in Soil	SPE-010	8880	Aroclor-1016 (PCB-1016)	EPA 8082	Pass
PCB in Soil	SPE-010	8885	Aroclor-1221 (PCB-1221)	EPA 8082	Pass
PCB in Soil	SPE-010	8890	Aroclor-1232 (PCB-1232)	EPA 8082	Pass
PCB in Soil	SPE-010	8895	Aroclor-1242 (PCB-1242)	EPA 8082	Pass
PCB in Soil	SPE-010	8900	Aroclor-1248 (PCB-1248)	EPA 8082	Pass
PCB in Soil	SPE-010	8905	Aroclor-1254 (PCB-1254)	EPA 8082	Pass
PCB in Soil	SPE-010	8910	Aroclor-1260 (PCB-1260)	EPA 8082	Pass
OrganoPhosphorus Pesticides	38151	7075	Azinphosmethyl	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7075	Azinphos-methyl	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7300	Chloropyrifos	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7390	Demeton O&S	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	7390	Demeton, (Mix of Isomers O:S)	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7410	Diazinon	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	7410	Diazinon	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	8610	Dichlorvos (DDVP)	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	8625	Disulfoton	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	8625	Disulfoton	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	8110	Fenchlorphos (Ronnell)	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7770	Malathion	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	7770	Malathion	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	7825	Parathion methyl	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7955	Parathion, ethyl	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	7825	Parathion, methyl	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	7985	Phorate	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	8110	Ronnell	EPA 8141A	Pass
OP/NP Pesticides in Soil	4280	8200	Stirophos (Tetrachlorvinphos)	EPA 8141A	Pass
OrganoPhosphorus Pesticides	38151	8200	Tetrachlorvinphos (Stirophos)	EPA 8141A	Pass
Herbicide Acids in Soil	38146	8655	2,4,5-T	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8650	2,4,5-TP	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8545	2,4-D	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8560	2,4-DB	EPA 8151A	Pass
Herbicides in Soil	4250	8600	3,5-Dichlorobenzoic acid	EPA 8151A	Pass
Herbicides in Soil	4250	8505	Acifluorfen	EPA 8151A	Pass
Herbicides in Soil	4250	8530	Bentazon	EPA 8151A	Pass
Herbicides in Soil	4250	8540	Chloramben	EPA 8151A	Pass
Herbicides in Soil	4250	8550	Dacthal diacid (DCPA)	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8555	Dalapon	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8595	Dicamba	EPA 8151A	Pass
Herbicide Acids in Soil	38146	8620	Dinoseb	EPA 8151A	Pass
Herbicide Acids in Soil	38146	6605	Pentachlorophenol	EPA 8151A	Pass
Herbicides in Soil	4250	6605	Pentachlorophenol	EPA 8151A	Pass
Herbicides in Soil	4250	8645	Picloram	EPA 8151A	Pass
RCRA Medium Level Volatiles in Soil	38199	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	5105	1,1,1,2-Tetrachloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5160	1,1,1-Trichloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5160	1,1,1-Trichloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	5160	1,1,1-Trichloroethane	EPA 8260B	Pass
Volatiles in Soil	4200	5160	1,1,1-Trichloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	5110	1,1,2,2-Tetrachloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5165	1,1,2-Trichloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5165	1,1,2-Trichloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	5165	1,1,2-Trichloroethane	EPA 8260B	Pass
Volatiles in Soil	4200	5165	1,1,2-Trichloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4630	1,1-Dichloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4630	1,1-Dichloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	4630	1,1-Dichloroethane	EPA 8260B	Pass
Volatiles in Soil	4200	4630	1,1-Dichloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4640	1,1-Dichloroethene	EPA 8260B	Pass
Volatiles in Soil	38084	4640	1,1-Dichloroethene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4640	1,1-Dichloroethylene	EPA 8260B	Pass
Volatiles in Soil	38084	4670	1,1-Dichloropropene	EPA 8260B	Pass
Volatiles in Soil	38084	5150	1,2,3-Trichlorobenzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5180	1,2,3-Trichloropropane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5180	1,2,3-Trichloropropane	EPA 8260B	Pass
Volatiles in Soil	38084	5180	1,2,3-Trichloropropane	EPA 8260B	Pass
Volatiles in Soil	4200	5180	1,2,3-Trichloropropane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5155	1,2,4-Trichlorobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5155	1,2,4-Trichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	5155	1,2,4-Trichlorobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5210	1,2,4-Trimethylbenzene	EPA 8260B	Pass
Volatiles in Soil	38084	5210	1,2,4-Trimethylbenzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4570	1,2-Dibromo-3-chloropropane	EPA 8260B	Pass
Volatiles in Soil	38084	4570	1,2-Dibromo-3-chloropropane	EPA 8260B	Pass
Volatiles in Soil	4200	4570	1,2-Dibromo-3-chloropropane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4570	1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4585	1,2-Dibromoethane	EPA 8260B	Pass
Volatiles in Soil	38084	4585	1,2-Dibromoethane	EPA 8260B	Pass
Volatiles in Soil	4200	4585	1,2-Dibromoethane (EDB)	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4585	1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260B	Pass
Volatiles in Soil	4200	4655	1,2-Dicahloropropane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4610	1,2-Dichlorobenzene	EPA 8260B	Pass

VOAs in Soil - Medium Level	SPE-002-H	4610	1,2-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4610	1,2-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4610	1,2-Dichlorobenzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4635	1,2-Dichloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4635	1,2-Dichloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	4635	1,2-Dichloroethane	EPA 8260B	Pass
Volatiles in Soil	4200	4635	1,2-Dichloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4655	1,2-Dichloropropane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4655	1,2-Dichloropropane	EPA 8260B	Pass
Volatiles in Soil	38084	4655	1,2-Dichloropropane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5215	1,3,5-Trimethylbenzene	EPA 8260B	Pass
Volatiles in Soil	38084	5215	1,3,5-Trimethylbenzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4615	1,3-Dichlorobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4615	1,3-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4615	1,3-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4615	1,3-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4660	1,3-Dichloropropane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4620	1,4-Dichlorobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4620	1,4-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4620	1,4-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4620	1,4-Dichlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4665	2,2-Dichloropropane	EPA 8260B	Pass
RCRA Ketones in Soil	38167	4410	2-Butanone (Methyl ethyl ketone)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4410	2-Butanone (Methyl ethyl ketone)	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4410	2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4500	2-Chloroethyl vinyl ether	EPA 8260B	Pass
Volatiles in Soil	4200	4500	2-Chloroethylvinylether	EPA 8260B	Pass
Volatiles in Soil	38084	4535	2-Chlorotoluene	EPA 8260B	Pass
RCRA Ketones in Soil	38167	4860	2-Hexanone	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4860	2-Hexanone	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4860	2-Hexanone	EPA 8260B	Pass
Volatiles in Soil	4200	4860	2-Hexanone	EPA 8260B	Pass
Volatiles in Soil	38084	4540	4-Chlorotoluene	EPA 8260B	Pass
RCRA Ketones in Soil	38167	4995	4-Methyl-2-pentanone	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4995	4-Methyl-2-pentanone	EPA 8260B	Pass
Volatiles in Soil	38084	4995	4-Methyl-2-pentanone	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4995	4-Methyl-2-pentanone (MIBK)	EPA 8260B	Pass
Volatiles in Soil	4200	4995	4-Methyl-2-pentanone (MIBK)	EPA 8260B	Pass
RCRA Ketones in Soil	38167	4315	Acetone	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4315	Acetone	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4315	Acetone	EPA 8260B	Pass
Volatiles in Soil	4200	4320	Acetonitrile	EPA 8260B	Pass
Volatiles in Soil	4200	4325	Acrolein	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4325	Acrolein (Propenal)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4375	Benzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4375	Benzene	EPA 8260B	Pass
Volatiles in Soil	38084	4375	Benzene	EPA 8260B	Pass
Volatiles in Soil	4200	4375	Benzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4385	Bromobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4385	Bromobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4385	Bromobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4385	Bromobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4390	Bromochloromethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4395	Bromodichloromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4395	Bromodichloromethane	EPA 8260B	Pass
Volatiles in Soil	38084	4395	Bromodichloromethane	EPA 8260B	Pass
Volatiles in Soil	4200	4395	Bromodichloromethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4400	Bromoform	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4400	Bromoform	EPA 8260B	Pass
Volatiles in Soil	38084	4400	Bromoform	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	49504	Bromomethane	EPA 8260B	Pass
Volatiles in Soil	38084	4950	Bromomethane	EPA 8260B	Pass
Volatiles in Soil	4200	4950	Bromomethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4450	Carbon disulfide	EPA 8260B	Pass
Volatiles in Soil	4200	4450	Carbon disulfide	EPA 8260B	Pass
Volatiles in Soil	38084	4450	Carbon disulfide	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4455	Carbon tetrachloride	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4455	Carbon tetrachloride	EPA 8260B	Pass
Volatiles in Soil	38084	4455	Carbon tetrachloride	EPA 8260B	Pass
Volatiles in Soil	4200	4455	Carbon tetrachloride	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4475	Chlorobenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4475	Chlorobenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4475	Chlorobenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4475	Chlorobenzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4485	Chloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4485	Chloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	4485	Chloroethane	EPA 8260B	Pass
Volatiles in Soil	4200	4485	Chloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4505	Chloroform	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4505	Chloroform	EPA 8260B	Pass
Volatiles in Soil	38084	4505	Chloroform	EPA 8260B	Pass
Volatiles in Soil	4200	4505	Chloroform	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4960	Chloromethane	EPA 8260B	Pass
Volatiles in Soil	38084	4960	Chloromethane	EPA 8260B	Pass
Volatiles in Soil	4200	4960	Chloromethane	EPA 8260B	Pass
Volatiles in Soil	4200	4645	cis-1,2-Dichloroethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4645	cis-1,2-Dichloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	4645	cis-1,2-Dichloroethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4645	cis-1,2-Dichloroethylene	EPA 8260B	Pass
Volatiles in Soil	4200	4680	cis-1,2-Dichloropropene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass
Volatiles in Soil	38084	4680	cis-1,3-Dichloropropene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4575	Dibromochloromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4575	Dibromochloromethane	EPA 8260B	Pass
Volatiles in Soil	38084	4575	Dibromochloromethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4595	Dibromomethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4595	Dibromomethane	EPA 8260B	Pass

Volatiles in Soil	38084	4595	Dibromomethane	EPA 8260B	Pass
Volatiles in Soil	4200	4595	Dibromomethane	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4625	Dichlorodifluoromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4625	Dichlorodifluoromethane	EPA 8260B	Pass
Volatiles in Soil	38084	4625	Dichlorodifluoromethane	EPA 8260B	Pass
Volatiles in Soil	4200	4625	Dichlorodifluoromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	9375	Di-isopropylether (DIPE)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4765	Ethyl benzene	EPA 8260B	Pass
Volatiles in Soil	38084	4765	Ethyl benzene	EPA 8260B	Pass
RCRA Oxygenates	38169	4770	Ethyl tert-butyl ether	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4765	Ethylbenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4765	Ethylbenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4835	Hexachlorobutadiene	EPA 8260B	Pass
Volatiles in Soil	4200	4835	Hexachlorobutadiene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4840	Hexachloroethane	EPA 8260B	Pass
Volatiles in Soil	38084	4840	Hexachloroethane	EPA 8260B	Pass
RCRA Oxygenates	38169	9375	Isopropyl ether	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4900	Isopropylbenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4900	Isopropylbenzene	EPA 8260B	Pass
Volatiles in Soil	38084	4900	Isopropylbenzene	EPA 8260B	Pass
Volatiles in Soil	4200	4900	Isopropylbenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5240	m+p-Xylene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4950	Methyl bromide (Bromomethane)	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4960	Methyl chloride (Chloromethane)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass
RCRA Oxygenates	38169	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass
Volatiles in Soil	38084	5000	Methyl tert-butyl ether (MTBE)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4975	Methylene chloride	EPA 8260B	Pass
Volatiles in Soil	38084	4975	Methylene chloride	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4975	Methylene chloride (Dichloromethane)	EPA 8260B	Pass
Volatiles in Soil	4200	4975	Methylene chloride (Dichloromethane)	EPA 8260B	Pass
Volatiles in Soil	4200	5000	Methyl-t-butylether (MTBE)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5005	Naphthalene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5005	Naphthalene	EPA 8260B	Pass
Volatiles in Soil	38084	5005	Naphthalene	EPA 8260B	Pass
Volatiles in Soil	38084	4435	n-Butyl benzene	EPA 8260B	Pass
RCRA Oxygenates	38169	5090	n-Propylbenzene	EPA 8260B	Pass
Volatiles in Soil	38084	5090	n-Propylbenzene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5250	o-Xylene	EPA 8260B	Pass
Volatiles in Soil	38084	4910	p-Isopropyl toluene	EPA 8260B	Pass
Volatiles in Soil	38084	4440	sec-Butyl benzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5100	Styrene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5100	Styrene	EPA 8260B	Pass
Volatiles in Soil	38084	5100	Styrene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4370	T-amylmethylether (TAME)	EPA 8260B	Pass
RCRA Oxygenates	38169	4370	tert-Amyl methyl ether	EPA 8260B	Pass
RCRA Oxygenates	38169	4420	tert-Butyl alcohol (t-Butanol)	EPA 8260B	Pass
Volatiles in Soil	38084	4445	tert-Butyl benzene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5115	Tetrachloroethene	EPA 8260B	Pass
Volatiles in Soil	38084	5115	Tetrachloroethene	EPA 8260B	Pass
Volatiles in Soil	4200	5115	Tetrachloroethene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5115	Tetrachloroethene (Perchloroethylene)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5140	Toluene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5140	Toluene	EPA 8260B	Pass
Volatiles in Soil	38084	5140	Toluene	EPA 8260B	Pass
Volatiles in Soil	4200	5140	Toluene	EPA 8260B	Pass
Volatiles in Soil	4200	4260	Total Xylenes	EPA 8260B	Pass
Volatiles in Soil	38084	5260	Total Xylenes	EPA 8260B	Pass
Volatiles in Soil	4200	5260	Total Xylenes	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4700	trans-1,2-Dichloroethene	EPA 8260B	Pass
Volatiles in Soil	38084	4700	trans-1,2-Dichloroethene	EPA 8260B	Pass
Volatiles in Soil	4200	4700	trans-1,2-Dichloroethene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4700	trans-1,2-Dichloroethylene	EPA 8260B	Pass
Volatiles in Soil	4200	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass
Volatiles in Soil	38084	4685	trans-1,3-Dichloropropene	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5170	Trichloroethene	EPA 8260B	Pass
Volatiles in Soil	38084	5170	Trichloroethene	EPA 8260B	Pass
Volatiles in Soil	4200	5170	Trichloroethene	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5170	Trichloroethene (Trichloroethylene)	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5175	Trichlorofluoromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5175	Trichlorofluoromethane	EPA 8260B	Pass
Volatiles in Soil	38084	5175	Trichlorofluoromethane	EPA 8260B	Pass
Volatiles in Soil	4200	5175	Trichlorofluoromethane	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5225	Vinyl acetate	EPA 8260B	Pass
Volatiles in Soil	4200	5225	Vinyl acetate	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5235	Vinyl chloride	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5235	Vinyl chloride	EPA 8260B	Pass
Volatiles in Soil	38084	5235	Vinyl chloride	EPA 8260B	Pass
Volatiles in Soil	4200	5235	Vinyl Chloride	EPA 8260B	Pass
VOAs in Soil - Medium Level	SPE-002-H	5260	Xylene, total	EPA 8260B	Pass
RCRA Medium Level Volatiles in Soil	38199	5260	Xylenes, total	EPA 8260B	Pass
RCRA Semi-Volatiles in Soil	38068	5155	1,2,4-Trichlorobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	4610	1,2-Dichlorobenzene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	4610	1,2-Dichlorobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	4615	1,3-Dichlorobenzene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	4615	1,3-Dichlorobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	4620	1,4-Dichlorobenzene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	4620	1,4-Dichlorobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6735	2,3,4,6-Tetrachlorophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6835	2,4,5-Trichlorophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6835	2,4,5-Trichlorophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6840	2,4,6-Trichlorophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6840	2,4,6-Trichlorophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6000	2,4-Dichlorophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6000	2,4-Dichlorophenol	EPA 8270C	Pass

Base Neutrals and Acids in Soil	4260	6130	2,4-Dimethylphenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6130	2,4-Dimethylphenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6175	2,4-Dinitrophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6175	2,4-Dinitrophenol	EPA 8270C	Pass
RCRA PAH's	38171	6185	2,4-Dinitrotoluene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6185	2,4-Dinitrotoluene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6005	2,6-Dichlorophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6005	2,6-Dichlorophenol	EPA 8270C	Pass
RCRA PAH's	38171	6190	2,6-Dinitrotoluene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6190	2,6-Dinitrotoluene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5795	2-Chloronaphthalene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5800	2-Chlorophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5800	2-Chlorophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6360	2-Methyl-4,6-Dinitrophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6385	2-Methylnaphthalene	EPA 8270C	Pass
RCRA PAH's	38171	6385	2-Methylnaphthalene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6385	2-Methylnaphthalene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6400	2-Methylphenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6460	2-Nitroaniline	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6490	2-Nitrophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6490	2-Nitrophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5945	3,3'-Dichlorobenzidine	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5945	3,3'-Dichlorobenzidine	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6405	3-Methylphenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6465	3-Nitroaniline	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6465	3-Nitroaniline	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6360	4,6-Dinitro-2-methylphenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5660	4-Bromophenyl phenyl ether	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5660	4-Bromophenyl phenyl ether	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5700	4-Chloro-3-methylphenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5700	4-Chloro-3-methylphenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5745	4-Chloroaniline	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5745	4-Chloroaniline	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5825	4-Chlorophenyl phenyl ether	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5825	4-Chlorophenyl-phenylether	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6410	4-Methylphenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6470	4-Nitroaniline	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6470	4-Nitroaniline	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6500	4-Nitrophenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6500	4-Nitrophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5500	Acenaphthene	EPA 8270C	Pass
RCRA PAH's	38171	5500	Acenaphthene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5500	Acenaphthene	EPA 8270C	Pass
Acenaphthylene in Soils	SPE-003	5505	Acenaphthylene	EPA 8270C	Pass
RCRA PAH's	38171	5505	Acenaphthylene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5505	Acenaphthylene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5545	Aniline	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5555	Anthracene	EPA 8270C	Pass
RCRA PAH's	38171	5555	Anthracene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5555	Anthracene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5575	Benzo(a)anthracene	EPA 8270C	Pass
RCRA PAH's	38171	5575	Benzo(a)anthracene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5575	Benzo(a)anthracene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5580	Benzo(a)pyrene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5585	Benzo(b)fluoranthene	EPA 8270C	Pass
RCRA PAH's	38171	5585	Benzo(b)fluoranthene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5585	Benzo(b)fluoranthene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass
RCRA PAH's	38171	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5600	Benzo(k)fluoranthene	EPA 8270C	Pass
RCRA PAH's	38171	5600	Benzo(k)fluoranthene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5600	Benzo(k)fluoranthene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5610	Benzoic Acid	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5630	Benzyl alcohol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5630	Benzyl alcohol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5670	Benzyl butyl phthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5670	Benzyl butyl phthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5760	bis(2-Chloroethoxy) methane	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5760	bis(2-Chloroethoxy) methane	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5765	bis(2-Chloroethyl) ether	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5765	bis(2-Chloroethyl) ether	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5780	bis(2-Chloroisopropyl) ether	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5780	bis(2-Chloroisopropyl) ether	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6255	bis(2-Ethylhexyl) phthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6065	bis(2-Ethylhexyl) phthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	14260	5680	Carbazole	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5680	Carbazole	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5855	Chrysene	EPA 8270C	Pass
RCRA PAH's	38171	5855	Chrysene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5855	Chrysene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5895	Dibenz(a,h)anthracene	EPA 8270C	Pass
RCRA PAH's	38171	5895	Dibenz(a,h)anthracene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5895	Dibenzo(a,h)anthracene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5905	Dibenzofuran	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5905	Dibenzofuran	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6070	Diethyl phthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6070	Diethyl phthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6135	Dimethyl phthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6135	Dimethyl phthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5925	Di-n-butyl phthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5925	Di-n-butylphthalate	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6200	Di-n-octyl phthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6200	Di-n-octylphthalate	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6265	Fluoranthene	EPA 8270C	Pass
RCRA PAH's	38171	6265	Fluoranthene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6265	Fluoranthene	EPA 8270C	Pass

Base Neutrals and Acids in Soil	4260	6270	Fluorene	EPA 8270C	Pass
RCRA PAH's	38171	6270	Fluorene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6270	Fluorene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6275	Hexachlorobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	4835	Hexachlorobutadiene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	4835	Hexachlorobutadiene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6285	Hexachlorocyclopentadiene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6285	Hexachlorocyclopentadiene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	4840	Hexachloroethane	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	4840	Hexachloroethane	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6315	Indeno (1,2,3-cd) pyrene	EPA 8270C	Pass
RCRA PAH's	38171	6315	Indeno (1,2,3-cd)pyrene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6315	Indeno(1,2,3-cd)pyrene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6320	Isophorone	EPA 8270C	Pass
RCRA PAH's	38171	6320	Isophorone	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6320	Isophorone	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6410	m/p-Cresol	EPA 8270C	Pass
RCRA PAH's	38171	5005	Naphthalene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5005	Naphthalene	EPA 8270C	Pass
RCRA PAH's	38171	5015	Nitrobenzene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5015	Nitrobenzene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	5015	Nitrobenzene (NB)	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6530	N-nitrosodimethylamine	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6530	N-Nitrosodimethylamine	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6545	N-nitrosodi-n-propylamine	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6545	N-Nitrosodi-n-propylamine	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6535	N-nitrosodiphenylamine	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6535	N-Nitrosodiphenylamine	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6400	o-Cresol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6605	Pentachlorophenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6615	Phenanthrene	EPA 8270C	Pass
RCRA PAH's	38171	6615	Phenanthrene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6615	Phenanthrene	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6625	Phenol	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6625	Phenol	EPA 8270C	Pass
Base Neutrals and Acids in Soil	4260	6665	Pyrene	EPA 8270C	Pass
RCRA PAH's	38171	6665	Pyrene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	6665	Pyrene	EPA 8270C	Pass
RCRA Semi-Volatiles in Soil	38068	5095	Pyridine	EPA 8270C	Pass
BNAs in Soil	SPE-003	5155	1,2,4-Trichlorobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	4610	1,2-Dichlorobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	4615	1,3-Dichlorobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	4620	1,4-Dichlorobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6835	2,4,5-Trichlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6840	2,4,6-Trichlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6000	2,4-Dichlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6130	2,4-Dimethylphenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6175	2,4-Dinitrophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6185	2,4-Dinitrotoluene (2,4-DNT)	EPA 8270C	Pass
BNAs in Soil	SPE-003	6005	2,6-Dichlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6190	2,6-Dinitrotoluene (2,6-DNT)	EPA 8270C	Pass
BNAs in Soil	SPE-003	5795	2-Chloronaphthalene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5800	2-Chlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6360	2-Methyl-4,6-dinitrophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6385	2-Methylnaphthalene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6400	2-Methylphenol (o-Cresol)	EPA 8270C	Pass
BNAs in Soil	SPE-003	6460	2-Nitroaniline	EPA 8270C	Pass
BNAs in Soil	SPE-003	6490	2-Nitrophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	5945	3,3'-Dichlorobenzidine	EPA 8270C	Pass
BNAs in Soil	SPE-003	6410	3+4-Methylphenol (m+p-Cresol)	EPA 8270C	Pass
BNAs in Soil	SPE-003	6405	3-Methylphenol (m-Cresol)	EPA 8270C	Pass
BNAs in Soil	SPE-003	6465	3-Nitroaniline	EPA 8270C	Pass
BNAs in Soil	SPE-003	5660	4-Bromophenyl phenyl ether	EPA 8270C	Pass
BNAs in Soil	SPE-003	5700	4-Chloro-3-methylphenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	5745	4-Chloroaniline	EPA 8270C	Pass
BNAs in Soil	SPE-003	5825	4-Chlorophenyl phenylether	EPA 8270C	Pass
BNAs in Soil	SPE-003	6410	4-Methylphenol (p-Cresol)	EPA 8270C	Pass
BNAs in Soil	SPE-003	6470	4-Nitroaniline	EPA 8270C	Pass
BNAs in Soil	SPE-003	6500	4-Nitrophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	5500	Acenaphthene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5505	Acenaphthylene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5545	Aniline	EPA 8270C	Pass
BNAs in Soil	SPE-003	5555	Anthracene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5595	Benzidine	EPA 8270C	Pass
BNAs in Soil	SPE-003	5575	Benzo(a)anthracene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5580	Benzo(a)pyrene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5585	Benzo(b)fluoranthene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5590	Benzo(g,h,i)perylene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5600	Benzo(k)fluoranthene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5610	Benzoic acid	EPA 8270C	Pass
BNAs in Soil	SPE-003	5630	Benzyl alcohol	EPA 8270C	Pass
BNAs in Soil	SPE-003	5760	bis(2-Chloroethoxy)methane	EPA 8270C	Pass
BNAs in Soil	SPE-003	5765	bis(2-Chloroethyl) ether	EPA 8270C	Pass
BNAs in Soil	SPE-003	5780	bis(2-Chloroisopropyl) ether	EPA 8270C	Pass
BNAs in Soil	SPE-003	6255	bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270C	Pass
BNAs in Soil	SPE-003	5670	Butyl benzyl phthalate	EPA 8270C	Pass
BNAs in Soil	SPE-003	5680	Carbazole	EPA 8270C	Pass
BNAs in Soil	SPE-003	5855	Chrysene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5895	Dibenz(a,h) anthracene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5905	Dibenzofuran	EPA 8270C	Pass
BNAs in Soil	SPE-003	6070	Diethyl phthalate	EPA 8270C	Pass
BNAs in Soil	SPE-003	6135	Dimethyl phthalate	EPA 8270C	Pass
BNAs in Soil	SPE-003	5925	Di-n-butyl phthalate	EPA 8270C	Pass
BNAs in Soil	SPE-003	6200	Di-n-octyl phthalate	EPA 8270C	Pass
BNAs in Soil	SPE-003	6265	Fluoranthene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6270	Fluorene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6275	Hexachlorobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	4835	Hexachlorobutadiene	EPA 8270C	Pass

BNAs in Soil	SPE-003	6285	Hexachlorocyclopentadiene	EPA 8270C	Pass
BNAs in Soil	SPE-003	4840	Hexachloroethane	EPA 8270C	Pass
BNAs in Soil	SPE-003	6315	Indeno(1,2,3-cd) pyrene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6320	Isophorone	EPA 8270C	Pass
BNAs in Soil	SPE-003	5005	Naphthalene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5015	Nitrobenzene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6530	n-Nitrosodimethylamine	EPA 8270C	Pass
BNAs in Soil	SPE-003	6545	n-Nitroso-di-n-propylamine	EPA 8270C	Pass
BNAs in Soil	SPE-003	6535	n-Nitrosodiphenylamine	EPA 8270C	Pass
BNAs in Soil	SPE-003	6605	Pentachlorophenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6615	Phenanthrene	EPA 8270C	Pass
BNAs in Soil	SPE-003	6625	Phenol	EPA 8270C	Pass
BNAs in Soil	SPE-003	6665	Pyrene	EPA 8270C	Pass
BNAs in Soil	SPE-003	5095	Pyridine	EPA 8270C	Pass
Base/Neutrals and Acids in Soil	727	5155	1,2,4-Trichlorobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	4610	1,2-Dichlorobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	4615	1,3-Dichlorobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	4620	1,4-Dichlorobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6835	2,4,5-Trichlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6840	2,4,6-Trichlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6000	2,4-Dichlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6130	2,4-Dimethylphenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6175	2,4-Dinitrophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6185	2,4-Dinitrotoluene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6005	2,6-Dichlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6190	2,6-Dinitrotoluene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5795	2-Chloronaphthalene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5800	2-Chlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6385	2-Methylnaphthalene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6400	2-Methylphenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6460	2-Nitroaniline	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6490	2-Nitrophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6410	3&4-Methylphenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5945	3,3'-Dichlorobenzidine	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6465	3-Nitroaniline	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6360	4,6-Dinitro-2-methylphenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5660	4-Bromophenyl-phenylether	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5700	4-Chloro-3-methylphenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5745	4-Chloroaniline	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5825	4-Chlorophenyl-phenylether	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6470	4-Nitroaniline	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6500	4-Nitrophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5500	Acenaphthene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5505	Acenaphthylene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5545	Aniline	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5555	Anthracene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5575	Benzo(a)anthracene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5580	Benzo(a)pyrene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5585	Benzo(b)fluoranthene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5590	Benzo(g,h,i)perylene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5600	Benzo(k)fluoranthene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5610	Benzoic acid	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5630	Benzyl alcohol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5760	bis(2-Chloroethoxy)methane	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5765	bis(2-Chloroethyl)ether	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5780	bis(2-Chloroisopropyl)ether	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6255	bis(2-Ethylhexyl)phthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5670	Butylbenzylphthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5855	Chrysene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5895	Dibenz(a,h)anthracene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5905	Dibenzofuran	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6070	Diethylphthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6135	Dimethylphthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5925	Di-n-butylphthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6200	Di-n-octylphthalate	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6265	Fluoranthene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6270	Fluorene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6275	Hexachlorobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	4835	Hexachlorobutadiene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6285	Hexachlorocyclopentadiene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	4840	Hexachloroethane	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6315	Indeno(1,2,3-cd)pyrene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6320	Isophorone	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5005	Naphthalene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5015	Nitrobenzene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6530	N-Nitrosodimethylamine	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6545	N-Nitroso-di-n-propylamine	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6535	N-Nitrosodiphenylamine	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6605	Pentachlorophenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6615	Phenanthrene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6625	Phenol	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	6665	Pyrene	EPA 8270D	Pass
Base/Neutrals and Acids in Soil	727	5095	Pyridine	EPA 8270D	Pass
Low-Level PAHs in Soil	722	5500	Acenaphthene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5505	Acenaphthylene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5555	Anthracene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5575	Benzo(a)anthracene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5580	Benzo(a)pyrene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5585	Benzo(b)fluoranthene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5590	Benzo(g,h,i)perylene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5600	Benzo(k)fluoranthene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5855	Chrysene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5895	Dibenz(a,h)anthracene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	6265	Fluoranthene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	6270	Fluorene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	6315	Indeno(1,2,3-cd)pyrene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	5005	Naphthalene	EPA 8270DSIM	Pass
Low-Level PAHs in Soil	722	6615	Phenanthrene	EPA 8270DSIM	Pass

Low-Level PAHs in Soil	722	6665	Pyrene	EPA 8270DSIM	Pass
Dioxin	SPE-016	9519	1,2,3,4,6,7,8,9-OCDD	EPA 8290	Pass
Dioxin	SPE-016	9516	1,2,3,4,6,7,8,9-OCDF	EPA 8290	Pass
Dioxin	SPE-016	9426	1,2,3,4,6,7,8-Hpccdd	EPA 8290	Pass
Dioxin	SPE-016	9420	1,2,3,4,6,7,8-Hpccdf	EPA 8290	Pass
Dioxin	SPE-016	9423	1,2,3,4,7,8,9-Hpccdf	EPA 8290	Pass
Dioxin	SPE-016	9453	1,2,3,4,7,8-Hxcdf	EPA 8290	Pass
Dioxin	SPE-016	9471	1,2,3,4,7,8-Hxcdd	EPA 8290	Pass
Dioxin	SPE-016	9456	1,2,3,6,7,8-Hxcdd	EPA 8290	Pass
Dioxin	SPE-016	9474	1,2,3,6,7,8-Hxcdf	EPA 8290	Pass
Dioxin	SPE-016	9459	1,2,3,7,8,9-Hxcdd	EPA 8290	Pass
Dioxin	SPE-016	9477	1,2,3,7,8,9-Hxcdf	EPA 8290	Pass
Dioxin	SPE-016	9549	2,3,4,7,8-Pecddf	EPA 8290	Pass
Dioxin	SPE-016	9606	2,3,7,8-TCDD	EPA 8290	Pass
Dioxin	SPE-016	9612	2,3,7,8-TCDF	EPA 8290	Pass
Dioxin	SPE-016	9444	Hpccdf, total	EPA 8290	Pass
Dioxin	SPE-016	9483	Hxcdf, total	EPA 8290	Pass
Dioxin	SPE-016	9606	PCDD + PCDF, total	EPA 8290	Pass
Dioxin	SPE-016	9993	PCDF, total	EPA 8290	Pass
Dioxin	SPE-016	9555	Pecdd, total	EPA 8290	Pass
Dioxin	SPE-016	9552	Pecdf, total	EPA 8290	Pass
Dioxin	SPE-016	9615	TCDD, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9540	1,2,3,7,8-Pecdd	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9543	1,2,3,7,8-Pecdf	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9480	2,3,4,6,7,8-Hxcdf	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9438	Hpccdd, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9444	Hpccdf, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9468	Hxcdd, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9483	Hxcdf, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9992	PCDD + PCDF, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9991	PCDD, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9993	PCDF, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9555	Pecdd, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9552	Pecdf, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9989	TCDD, total	EPA 8290	Pass
Dioxins and Furans in Soil	SPE-016	9615	TCDF, total	EPA 8290	Pass
Herbicides in Soil	4250	8655	2,4,5-T	EPA 8321A	Pass
Herbicides in Soil	4250	8650	2,4,5-TP (silvex)	EPA 8321A	Pass
Herbicides in Soil	4250	8545	2,4-D	EPA 8321A	Pass
Herbicides in Soil	4250	8560	2,4-DB	EPA 8321A	Pass
RCRA Carbamates	38158	7710	3-Hydroxycarbofuran	EPA 8321A	Pass
RCRA Carbamates	38158	7015	Aldicarb sulfone	EPA 8321A	Pass
RCRA Carbamates	38158	7020	Aldicarb sulfoxide	EPA 8321A	Pass
RCRA Carbamates	38158	8080	Baygon (Propoxur)	EPA 8321A	Pass
RCRA Carbamates	38158	7195	Carbaryl	EPA 8321A	Pass
RCRA Carbamates	38158	7205	Carbofuran	EPA 8321A	Pass
Herbicides in Soil	4250	8555	Dalapon	EPA 8321A	Pass
Herbicides in Soil	4250	8595	Dicamba	EPA 8321A	Pass
Herbicides in Soil	4250	8605	Dichloroprop	EPA 8321A	Pass
Herbicides in Soil	4250	8620	Dinoseb	EPA 8321A	Pass
RCRA Carbamates	38158	9384	Dioxacarb	EPA 8321A	Pass
RCRA Carbamates	38158	7505	Diuron	EPA 8321A	Pass
Herbicides in Soil	4250	7775	MCPA	EPA 8321A	Pass
Herbicides in Soil	4250	7780	MCPP	EPA 8321A	Pass
RCRA Carbamates	38158	7800	Methiocarb	EPA 8321A	Pass
RCRA Carbamates	38158	8025	Promecarb	EPA 8321A	Pass
Nitroaromatics/Nitroamines in Soil	4420	6885	1,3,5-Trinitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	9651	2,4,6-Trinitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	6185	2,4-Dinitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	6190	2,6-Dinitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	9306	4-Amino-2,6-dinitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	9513	4-Nitrotoluene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	3740	HMX	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	6900	Nitrobenzene	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	3630	RDX	EPA 8330	Pass
Nitroaromatics/Nitroamines in Soil	4420	6415	Tetryl	EPA 8330	Pass
RCRA Nitroaromatics in Soil	38155	6885	1,3,5-Trinitrobenzene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	6160	1,3-Dinitrobenzene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9651	2,4,6-Trinitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	6185	2,4-Dinitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	6190	2,6-Dinitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9303	2-Amino-4,6-dinitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9507	2-Nitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9510	3-Nitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9306	4-Amino-2,6-dinitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9513	4-Nitrotoluene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9522	HMX	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	5015	Nitrobenzene	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	9432	RDX	EPA 8330A	Pass
RCRA Nitroaromatics in Soil	38155	6415	Tetryl	EPA 8330A	Pass
Nitroaromatics in Soil	38155	6885	1,3,5-Trinitrobenzene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	6160	1,3-Dinitrobenzene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9651	2,4,6-Trinitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9185	2,4-Dinitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	6190	2,6-Dinitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9303	2-Amino-4,6-dinitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9507	2-Nitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9510	3-Nitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9306	4-Amino-2,6-dinitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9513	4-Nitrotoluene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9522	HMX	EPA 8330B	Pass
Nitroaromatics in Soil	38155	5015	Nitrobenzene	EPA 8330B	Pass
Nitroaromatics in Soil	38155	9432	RDX	EPA 8330B	Pass
Nitroaromatics in Soil	38155	6415	Tetryl	EPA 8330B	Pass
RCRA Cyanide	55105	1645	Cyanide	EPA 9010B	Pass
Cyanide in Soil	4130	1645	Total Cyanide	EPA 9010B	Pass
RCRA Cyanide	55105	1645	Cyanide	EPA 9014	Pass
Cyanide in Soil	4130	1645	Total Cyanide	EPA 9014	Pass

RCRA Corrosivity - pH Determination	55127	1625	Corrosivity	EPA 9045C	Pass
pH/Corrosivity in Soil	4140	1625	Corrosivity (pH)	EPA 9045C	Pass
RCRA Corrosivity - pH Determination	55127	1625	Corrosivity	EPA 9045D	Pass
Anions in Soil	4160	1540	Bromide	EPA 9056	Pass
RCRA Anions	55141	1540	Bromide (Br)	EPA 9056	Pass
Anions in Soil	4160	1575	Chloride	EPA 9056	Pass
RCRA Anions	55141	1575	Chloride (Cl)	EPA 9056	Pass
Anions in Soil	4160	1730	Fluoride	EPA 9056	Pass
RCRA Anions	55141	1730	Fluoride (F)	EPA 9056	Pass
RCRA Anions	55141	1810	Nitrate as N (NO3- as N)	EPA 9056	Pass
Anions in Soil	4160	1810	Nitrate Nitrogen as N	EPA 9056	Pass
Anions in Soil	4160	1870	Orthophosphate as P	EPA 9056	Pass
RCRA Anions	55141	1870	Phosphate as P (PO43- as P)	EPA 9056	Pass
Anions in Soil	4160	2000	Sulfate	EPA 9056	Pass
RCRA Anions	55141	2000	Sulfate (SO42-)	EPA 9056	Pass
Anions in Soil	SPE-013	1540	Bromide	EPA 9056A	Pass
Anions in Soil	SPE-013	1575	Chloride	EPA 9056A	Pass
Anions in Soil	SPE-013	1730	Fluoride	EPA 9056A	Pass
Anions in Soil	SPE-013	1810	Nitrate as N	EPA 9056A	Pass
Anions in Soil	SPE-013	1820	Nitrate+nitrite as N	EPA 9056A	Pass
Anions in Soil	SPE-013	1840	Nitrite as N	EPA 9056A	Pass
Anions in Soil	SPE-013	1870	Orthophosphate as P	EPA 9056A	Pass
Anions in Soil	SPE-013	2000	Sulfate	EPA 9056A	Pass

## Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270  
Nashville, TN 37228  
Marcia K. McGinnity  
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In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

### Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Di-isopropyl ether
GC/MS	8260B	ETBE
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	t-Butyl alcohol
GC/MS	8260B	tert-Amyl methyl ether
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	3,5-Dinitroaniline
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	8011	1,2-Dibromoethane (EDB)
GC/ECD	8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7470A	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
IC	300.0	Chloride
IC	300.0	Fluoride

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
IC	300.0	Nitrate
IC	300.0	Nitrite
IC	300.0	Sulfate
IC	9056A	Chloride
IC	9056A	Fluoride
IC	9056A	Nitrate
IC	9056A	Nitrite
IC	9056A	Sulfate
Titration	SM 2320B 20th ed.	Alkalinity
ISE	SM 4500 B, D, 20th ed.	Ammonia
UV/Vis	7196A	Hexavalent Chromium
Colorimetric	353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th edition	Total Phosphorus
UV/Vis	SM 4500 PE, 20th edition	Ortho-Phosphorus
TOC	9060A/SM5310C, 20 <sup>th</sup> edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th edition	TDS
Colorimetric	9012A/B	Cyanide
Physical	1010A	Ignitability
Physical	9095B	Paint Filter
Probe	9040B/C	pH
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Preparation	1311	TCLP
Preparation	3005A	Metals digestion
Preparation	3010A	Metals digestion
Preparation	3510C	Organics Liquid Extraction
Preparation	5030A/B	Purge and Trap Water

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBd)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7471A/B	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
UV/Vis	7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	9034	Sulfide
Probe	9045D	pH
Preparation	Method	Type
Preparation	1311	TCLP
Preparation	1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	3050B	Metals Digestion
Preparation	3546	Organics Microwave Extraction
Preparation	3541	Organics Soxhlet Extraction
Preparation	3550B	Organics Sonication
Preparation	SM 2540B 20th edition	Percent Solids (Percent Moisture)
Preparation	5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: \_\_\_\_\_



R. Douglas Leonard  
Chief Technical Officer

Date: November 30, 2009

Issued: 11/30/09

SOP Title:

**BNA & Pesticide/PCBs & TPH NON-  
AQUEOUS MATRIX (MICROWAVE  
EXTRACTION) USING SW-846 METHOD  
3546**

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SOP NUMBER:

**SOP-343**

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REVISION NUMBER:

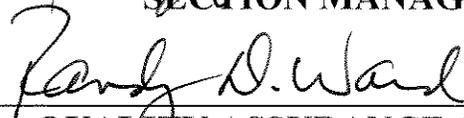
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APPROVED BY:

  
SECTION MANAGER

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QUALITY ASSURANCE OFFICER

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EFFECTIVE DATE:

**08/01/09**

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DATE OF LAST REVIEW:

**08/01/09**

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**BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX  
(Microwave Extraction)  
Using SW846 METHOD 3546**

**1. SCOPE AND APPLICATION**

- a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

**2. SUMMARY**

- a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

**3. INTERFERENCES**

- a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
- b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
- c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**APPARATUS AND MATERIALS**

- d. Stainless Steel spatula
- e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
- f. Microwave extraction Teflon tubes, capacity approximately 75mL
- g. Suitable Teflon cap and screw-top lid
- h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
- i. Vial – 2mL clear with Teflon-lined screw cap
- j. Vial – 12mL clear with Teflon-lined screw cap
- k. Syringe – 1mL, 500uL
- l. Pasteur pipet – 9” length
- m. Pasteur pipet bulb
- n. Labels – Dymo
- o. Aluminum foil – heavy duty
- p. Nitrogen tank – equipped with pressure regulator
- q. TurboVap Concentrator with 200mL concentrator tubes
- r. Teflon funnels for pouring off
- s. Balance – capable of weighing to 0.1grams
- t. Aluminum pie pans for mixing samples
- u. Filter paper – 185mm

#### 4. REAGENTS

- a. Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
- b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
- c. Hexane – suitable for spectrophotometry and gas chromatography (Fisher #H303-4)
- d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
  - i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0ug/mL BN Surrogate spiking solution.)**
  - ii. **BNA Spiking Solution #1 & #2 (100 ug/mL)** – The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL PAH spiking solution.) The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
  - iii. **TCMX/DCB (2,4,5,6-Tetrachloro-metaxylene/Decachlorobiphenyl) Surrogate solution** is prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
  - iv. **PCB Spiking Solution** – Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
  - v. **Pesticide Spiking Solution** – A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
  - vi. **TPH Surrogate** – Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
  - vii. **TPH Spike** – A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

## 5. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
- b. Samples are preserved by cooling to 4°C.
- c. Holding time is 14 days from collection date to extraction.

## 6. PROCEDURE

- a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
  - i. Each day a backlog is generated in ELEMENT providing all relevant sample information, including samples numbers and respective analysis required.
  - ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
  - iii. Check the backlog throughout the day to re-evaluate priority if needed.
- b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
- c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
- d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
- e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container**

- f. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.
- g. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
- h. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.

NOTE: Surrogate and spike should be added just prior to extraction.

- i. Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH samples will need 1.0 mL of appropriate.

For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**

For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:

**Pesticide and PCB** - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

**Pesticide only** – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

**PCB only** - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na<sub>2</sub>SO<sub>4</sub>.

- j. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
- k. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.
- l. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
- m. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

For 1-15 samples:

Max power: 800W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

For 16-40 samples:

Max power: 1600W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

- n. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
- o. Transfer the extract to a pre-rinsed turbo vap tube by first passing through a funnel with P4 filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with Methylene Chloride and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.
- p. Now concentrate the extract to 1.0mL using the turbovap concentrator.
  - i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 50-55°C. The pressure target range should be about 20-25 psi.
  - ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
  - iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- q. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

## 7. DOCUMENTATION OF CAPABILITY (DOC)

- a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## 8. WASTE MANAGEMENT AND POLLUTION PREVENTION

- a. Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.
- b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **9. METHOD PERFORMANCE**

- a. Refer to SOP-201 SOP-211 and SOP-219 for method performance.

## **10. REFERENCES**

- a. EPA Methods SW-846, Method 3546

## **11. DEFINITIONS**

- a. Refer to SOP-431 for definitions.

## **12. HEALTH AND SAFETY**

- a. Wear appropriate personal protection equipment when working with chemicals or samples.
- b. Use the lab hoods when working with solvents.
- c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

## GCMSrpDLInfo

	Method	Analyte	MRL/LOQ	LOD	MDL/DL	Units	QC Limits
Water	8270C/D low	1-Methylnaphthalene	0.200	0.100	0.0500	ug/L	35-131
Water	8270C/D low	2-Methylnaphthalene	0.200	0.100	0.0500	ug/L	36-121
Water	8270C/D low	Acenaphthene	0.200	0.100	0.0500	ug/L	41-132
Water	8270C/D low	Acenaphthylene	0.200	0.100	0.0500	ug/L	43-140
Water	8270C/D low	Anthracene	0.200	0.100	0.0500	ug/L	50-139
Water	8270C/D low	Benzo (a) anthracene	0.200	0.100	0.0500	ug/L	58-141
Water	8270C/D low	Benzo (a) pyrene	0.200	0.100	0.0500	ug/L	31-142
Water	8270C/D low	Benzo (b) fluoranthene	0.200	0.100	0.0500	ug/L	42-156
Water	8270C/D low	Benzo (g,h,i) perylene	0.200	0.100	0.0500	ug/L	12-171
Water	8270C/D low	Benzo (k) fluoranthene	0.200	0.100	0.0500	ug/L	49-165
Water	8270C/D low	Chrysene	0.200	0.100	0.0500	ug/L	51-155
Water	8270C/D low	Dibenz (a,h) anthracene	0.200	0.100	0.0500	ug/L	28-153
Water	8270C/D low	Fluoranthene	0.200	0.100	0.0500	ug/L	47-158
Water	8270C/D low	Fluorene	0.200	0.100	0.0500	ug/L	40-140
Water	8270C/D low	Indeno (1,2,3-cd) pyrene	0.200	0.100	0.0500	ug/L	20-167
Water	8270C/D low	Naphthalene	0.200	0.100	0.0500	ug/L	39-125
Water	8270C/D low	Phenanthrene	0.200	0.100	0.0500	ug/L	46-144
Water	8270C/D low	Pyrene	0.200	0.100	0.0500	ug/L	39-158
Water	8270C/D low	2-Fluorobiphenyl	-	-	-	ug/L	34-167
Water	8270C/D low	Terphenyl-d14	-	-	-	ug/L	34-167
Solid	8270C/D Low	1-Methylnaphthalene	6.67	3.33	1.67	ug/Kg	30-111
Solid	8270C/D Low	2-Methylnaphthalene	6.67	3.33	1.67	ug/Kg	30-111
Solid	8270C/D Low	Acenaphthene	6.67	3.33	1.67	ug/Kg	28-110
Solid	8270C/D Low	Acenaphthylene	6.67	3.33	1.67	ug/Kg	23-126
Solid	8270C/D Low	Anthracene	6.67	3.33	1.67	ug/Kg	28-136
Solid	8270C/D Low	Benzo (a) anthracene	6.67	3.33	1.67	ug/Kg	31-146
Solid	8270C/D Low	Benzo (a) pyrene	6.67	3.33	1.67	ug/Kg	28-128
Solid	8270C/D Low	Benzo (b) fluoranthene	6.67	3.33	1.67	ug/Kg	30-139
Solid	8270C/D Low	Benzo (g,h,i) perylene	6.67	3.33	1.67	ug/Kg	21-149
Solid	8270C/D Low	Benzo (k) fluoranthene	6.67	3.33	1.67	ug/Kg	42-129
Solid	8270C/D Low	Chrysene	6.67	3.33	1.67	ug/Kg	39-134
Solid	8270C/D Low	Dibenz (a,h) anthracene	6.67	3.33	1.67	ug/Kg	30-138
Solid	8270C/D Low	Fluoranthene	6.67	3.33	1.67	ug/Kg	30-142
Solid	8270C/D Low	Fluorene	6.67	3.33	1.67	ug/Kg	27-116
Solid	8270C/D Low	Indeno (1,2,3-cd) pyrene	6.67	3.33	1.67	ug/Kg	17-164
Solid	8270C/D Low	Naphthalene	6.67	3.33	1.67	ug/Kg	29-106
Solid	8270C/D Low	Phenanthrene	6.67	3.33	1.67	ug/Kg	32-127
Solid	8270C/D Low	Pyrene	6.67	3.33	1.67	ug/Kg	28-130
Solid	8270C/D Low	2-Fluorobiphenyl	-	-	-	ug/Kg	14-129
Solid	8270C/D Low	Terphenyl-d14	-	-	-	ug/Kg	14-129