

N62578.AR.002609
NCBC DAVISVILLE
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DRAFT FINAL SAMPLING AND ANALYSIS PLAN (FIELD SAMPLING AND QUALITY
ASSURANCE PROJECT PLAN AUGUST 2010) FOR CONFIRMATORY SAMPLING AND
DRAIN LINE INVESTIGATION CED AREA/QDC OUTFALL 001 NCBC DAVISVILLE RI (DRAFT
ACTING AS FINAL)
8/1/2010
TETRA TECH

**DRAFT FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
August 2010**

**Confirmatory Sampling and Drain Line Investigation
CED Area/QDC Outfall 001
Former Naval Construction Battalion Center Davisville
North Kingstown, Rhode Island**

**Prepared for:
Naval Facilities Engineering Command
Mid-Atlantic Division
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**Prepared under:
Contract Number N62472-03-D-0057
“CLEAN” Contract Task Order No. 19**

Project-Specific Sampling and Analysis Plan
Site Name: CED Area/QDC Outfall 001, NCBC Davleville
Project Name: Confirmatory Sampling and Drain Line Investigation
Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
Document No.: WR200578DF
Revision Number: 0
Revision Date: August 2010

SAP Worksheet #1 -- Approval Page
(JFP-QAPP Manual Section 2.1)

Document Title: Draft Final Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), August 2010, Confirmatory Sampling and Drain Line Investigation, CED Area/ QDC Outfall 001, Former NCBC Davleville

Lead Organization: Naval Facilities Engineering Command Mid-Atlantic

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Preparation Date (Day/Month/Year): August 2010

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Signature/Date
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EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) presents the technical approach for an investigation to be conducted by Tetra Tech NUS, Inc. (Tetra Tech) on behalf of the Navy to assess the presence and nature of contamination in soils and sediment at the former Naval Construction Battalion Center (NCBC) Davisville in North Kingstown, Rhode Island. This SAP was prepared under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract No. N62472-03-D-0057, Contract Task Order (CTO) 19 in accordance with the Navy's Uniform Federal Policy for Sampling and Analysis Plans (UFP-SAP) guidance to ensure that environmental data collected is scientifically sound, of known and documented quality, and suitable for the intended purposes.

The former NCBC Davisville is located in the town of North Kingstown, Rhode Island. The Former NCBC Davisville was primarily used for training Navy Construction Battalion "Seabees" in construction operations, and as storage and freight yards for construction materials. The facility was closed by the Navy on April 1, 1994.

The Navy's Former Construction Equipment Department (CED) was located on a parcel of land bounded by Seabee Avenue to the west, Perimeter Road to the north, Davisville Road to the south, and light wooded vegetation to the east (Figure 10-1). The CED area has been the subject of several environmental response actions since closure of the NCBC. These actions have been taken to address the following release areas:

- Study Area 01 (CED Drum Storage Area),
- Site 02 (CED Battery Acid Disposal Area),
- Site 03 (CED Solvent Disposal Area), and
- Study Area 04 (CED Asphalt Disposal Area).

The strategy for closure of these sites/study areas is to address them together as one operable unit. Therefore, these areas are collectively referred to as the "CED Area".

Quonset Development Corporation (QDC) Outfall 001 is located in the central portion of the former NCBC Davisville facility, to the east of the CED Area and to the west of Allen Harbor (Figure 10-1). QDC Outfall 001 is located near the intersection of Marine Road and Sanford Road, behind a chain-link fence that surrounds the parking area for recreational users of Allen Harbor Landfill and Calf Pasture Point (Figure 10-2). Undeveloped wetlands are present to the east of the outfall.

Based on a review of historical as-built drawings of drainage systems at the former NCBC, Outfall 001 is the discharge point for an underground drainage line that originated from the former Building 224.

Building 224 was part of the CED Area located approximately 1,000 feet to the southwest of the outfall (Figure 10-3). Building 224 was used by the Navy as a vehicle maintenance and truck washing facility. Contaminated materials from these activities or other historical activities at the Former CED may have migrated into or been disposed of into the Building 224 drainage system and discharged into the outfall area. The Navy has conducted several removal actions at the CED Area to address contamination encountered in surface and subsurface soils. The contaminants associated with these removals included waste oils, solvents, lead, PAHs, PCBs, and petroleum hydrocarbons.

In the summer of 2008 during QDC's storm water outfall maintenance activities, QDC excavated soil that was present downstream from the Outfall 001 drain pipe outlet. During this excavation, QDC observed stained soils and olfactory evidence of contamination. QDC stockpiled this soil adjacent to the outfall (Figure 2) and contacted the Navy. At the BRAC Cleanup Team (BCT) meeting on September 25, 2008, the Navy agreed to characterize and dispose of the soils that were stockpiled adjacent to the outfall. In December 2008 a composite soil sample was collected from the stockpile for the purpose of characterizing this material for off-site disposal. Analytical results from the soil stockpile sample indicated the presence of TPH (>10,000 mg/kg), VOCs, PCBs, PAHs, and metals, some of which exceeded state or federal direct exposure criteria or risk-based screening criteria. In late December 2008, approximately 23 tons of soil were transported for off-site disposal at an approved landfill.

It is the project team's assumption that the source of contamination to the soils downstream of the drain pipe outlet in the excavation area was the discharge from the drainage pipe that formerly originated from Building 224. Chemicals used during historical truck maintenance activities (and other historical activities that occurred in and around the Building 224 area) probably entered the drainage system and were transported along the length of the pipe, discharging into soils in the drainage swale downstream from the pipe and potentially migrating with the flow of surface water further downstream toward the wetland area.

In addition to the potential for contamination in the outfall area soil and sediment, there is a potential for contamination in subsurface soil adjacent to the underground drain pipe. Given the age of the drain pipe, it is reasonable to expect that it may have been compromised along its length and some contaminated materials may have leaked into the soils adjacent to the pipe, thereby potentially contaminating nearby subsurface soils and downgradient groundwater.

Access to the outfall area is currently restricted by the chain link fence that surrounds the parking area that is located along Sanford Road. Persons currently accessing the site include trespassers, although there are recreational areas in close proximity to the outfall. Given that the future land use is unknown, it is customary to evaluate use of a property as residential and recreational. Therefore, potential future human receptors include residents and recreational users, in addition to industrial and construction workers and trespassers. Ecological receptors include animal and plant species that could be affected by the contaminants present in environmental media. Human and ecological receptors may be affected by

contaminants in the excavation area, and human receptors may be affected by subsurface soil in the area alongside the drain line. Ecological receptors typically are not exposed to subsurface media (greater than 3 feet bgs).

The Navy will perform a surface and subsurface soil, sediment, and residual material investigation to address the following environmental questions:

- Are there chemicals in soils remaining in the excavation area and sediments in the wetlands at concentrations that may present a risk to human health or the environment?
- Are there areas of compromised integrity of the pipe? If so, are there contaminants in the subsurface soil along the drain line and, if so, are the contaminants detected at concentrations that may present a risk to human health?
- Is residual material present in the drain pipe or in areas where the drain pipe has been compromised providing a continuing source of contamination to the area downstream from the drain pipe outlet and the wetland area?

To address these questions, the following field investigations and decisions are proposed:

- Collection of soil samples from the previously-excavated area to determine whether or not there are contaminant concentrations in soils remaining in the excavation area that exceed human health and ecological risk-based screening criteria.
- Collection of sediment samples from the wetlands downgradient from the outfall to determine whether or not there are contaminant concentrations in sediments that exceed human health and ecological risk-based screening criteria.
- A reconnaissance of the interior of the drain pipe to document any areas where the integrity of the pipe has been compromised, in order to identify potential areas of release of contaminants to the soils adjacent to the pipe.
- Investigation of the residual material that is present in the drain pipe, near the pipe outlet, and from compromised portions of the pipe (should residual materials be encountered) to determine whether or not the material in the pipe is a continuing source of contamination to soils downstream from the pipe outlet and sediment in the wetland area.

- Investigation of subsurface soil located alongside the drain pipe in order to characterize potential contamination in the soil due to the release of residual material from the pipe and to determine whether or not the subsurface soil contamination concentrations exceed human health risk-based criteria.

Field investigations are planned for 2010. The data collected during the investigation will be presented to the BCT in a letter report documenting the investigations completed, the findings of the investigations, and a comparison of analytical data to risk-based screening criteria, and recommendations for additional investigations or no further action, as necessary.

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TABLES

NUMBER

- 10-1 Soil Stockpile Sample Analytical Results Exceeding RIDEM Soil Residential DECs, RSLs, and/or Selected Ecological Soil or Sediment Screening Levels

FIGURES

NUMBER

- 10-1 Site Locus Map
- 10-2 CED Area Site Plan
- 10-3 Former Building 224 Drain Line and QDC Outfall Location
- 17-1 Proposed Soil/Sediment Sampling Locations
- 17-2 Proposed Test Pitting and Sampling Locations

REFERENCES

APPENDICES

- A Site Photographs
- B Soil Stockpile Sample Analytical Data
- C Field Documentation Forms
- D Sampling and Related SOPs
- E Analytical Specification
- F Project-Specific Field Task Procedures
- G Laboratory SOPs
- H Determination of Project Action Limits

ACRONYMS

ANSI/ASQ	American National Standards Institute/American Society for Quality
ASTM	American Society for Standards and Materials
BCT	BRAC Closure Team
BEC	BRAC Environmental Coordinator
BRAC	Base Realignment and Closure
CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
DL	Detection Limit
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPS	Global Positioning System
GW	Ground Water
HHRA	Human Health Risk Assessment
HI	Hazard Index
HQ	Hazard Quotient
ICP	Inductively Coupled Plasma
IR	Installation Restoration (Navy)
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
LOD	Limit of Detection
LOQ	Limit of Quantitation
LUC	Land Use Control
MCL	Maximum Contaminant Level
MPC	Measurement Performance Criteria
MQO	Measurement Quality Objectives
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSL	Mean Sea Level
MSR	Management Systems Review
NCP	National Contingency Plan
NPL	National Priorities List
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCBs	Polychlorinated Biphenyls
PDF	Portable Document Format
PID	Photoionization Detector
PM	Project Manager
PQL	Project Quantitation Limit
PQOs	Project Quality Objectives
PRQL	Project-Required Quantitation Limit
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control

ACRONYMS (cont.)

QMP	Quality Management Plan
QS	Quality System
QSM	Quality Systems Manual
RAO	Remedial Action Objective
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RIDEM	Rhode Island Department of Environmental Management
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RTM	Remedial Technical Manager
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
SQLs	Sample Quantitation Limits
SVOC	Semivolatile Organic Compounds
SW	Surface Water
TBD	To Be Determined
TSA	Technical Systems Audit
UFP	Uniform Federal Policy
VOC	Volatile Organic Compounds

SAP Worksheet #2 – SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: QDC Outfall 001, Former NCBC Davisville, North Kingstown, Rhode Island

Operable Unit: Not applicable

Contractor Name: Tetra Tech NUS, Inc. (Tetra Tech)

Contract Number: N62472-03-D-0057

Contract Title: NAVFAC Mid-Atlantic CLEAN

Work Assignment Number (optional): CTO 19

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (IDQTF 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)*.

2. Identify regulatory program: National Contingency Plan (NCP); 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
<u>Draft Outfall 001 Soil Characterization & Removal</u>	<u>January 21, 2009</u>
<u>Navy/Tetra Tech Scoping Discussion</u>	<u>March 12, 2009</u>
<u>EPA/RIDEM Comments and Navy Responses on Draft SAP for Confirmatory Sampling and Drain Line Investigation at QDC Outfall 001</u>	<u>Aug-Oct 2009</u>

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

Title (Author)	Date
<u>Draft Outfall 001 Soil Characterization and Removal</u>	<u>January 21, 2009</u>
_____	_____
_____	_____

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

<u>Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic – lead organization</u>
<u>U. S. Environmental Protection Agency (EPA), Region I – regulator</u>
<u>Rhode Island Department of Environmental Management (RIDEM) – regulator</u>
<u>Quonset Development Corporation – local redevelopment authority</u>
<u>Town of North Kingstown – property owner</u>

7. Lead organization

<u>NAVFAC Mid-Atlantic</u>

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:

<u>None</u>

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

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
David Barney	Navy BEC	BRAC PMO Northeast NAVFAC MIDLANT	(617) 753-4656	David.A.Barney@navy.mil	NA
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David Barclift	Navy Technical Support	BRAC PMO Northeast NAVFAC Atlantic	(215) 897-4913	David.Barclift@navy.mil	NA
Christine Williams	EPA RPM	EPA Region I	(617) 918-1384	Williams.Christine@epa.gov	NA
Kathleen Campbell	EPA Contractor	CDW Consultants		kcampbell@cdwconsultants.com	NA
Rich Gottlieb	RIDEM RPM	RIDEM	(401) 222-2797	Richard.Gottlieb@dem.ri.gov	NA
Steven King	Managing Director	Quonset Development Corporation		95 Cripe Street North Kingstown, RI 02852	NA
Jon Reiner	Planning Director	Town of North Kingstown		80 Boston Neck Road North Kingstown, RI 02852-5762	NA
Stephen Vetere	Tetra Tech Engineer	Tetra Tech, Inc.	(978) 474-8444	Stephen.Vetere@tetrattech.com	NA
Scott Anderson	Tetra Tech Project Manager (PM)	Tetra Tech, Inc.	(412) 921-8608	Scott.Anderson@tetrattech.com	NA
Lee Ann Sinagoga	Tetra Tech Risk Assessor	Tetra Tech, Inc.	(412) 921-8887	LeeAnn.Sinagoga@tetrattech.com	NA
Lucy Guzman	Tetra Tech Project Chemist	Tetra Tech, Inc.	(978) 474-8416	Lucy.Guzman@tetrattech.com	NA
Michael Alroy	Tetra Tech Field Operations Leader	Tetra Tech, Inc.	(978) 474-8450	Michael.Alroy@tetrattech.com	NA
Tom Johnston	Tetra Tech Project Quality Assurance Manager (QAM)	Tetra Tech, Inc.	(412) 921-8615	Tom.Johnston@tetrattech.com	NA
Matt Soltis	Tetra Tech Health and Safety Manager (HSM)	Tetra Tech, Inc.	(412) 921-8912	Matt.Soltis@tetrattech.com	NA
Glenn Wagner	Administrative Record	Tetra Tech, Inc.	(412) 320-2211	Glenn.Wagner@tetrattech.com	NA
Ed Lawler	Laboratory Operations Manager	Mitkem Laboratories	(401) 732-3400	elawler@mitkem.com	NA

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
Project Name: Confirmatory Sampling and Drain Line Investigation
Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
Document No.: W5209575DF
Revision Number: 0
Revision Date: August 2010

SAP Worksheet #4 – Project Personnel Sign-Off Sheet

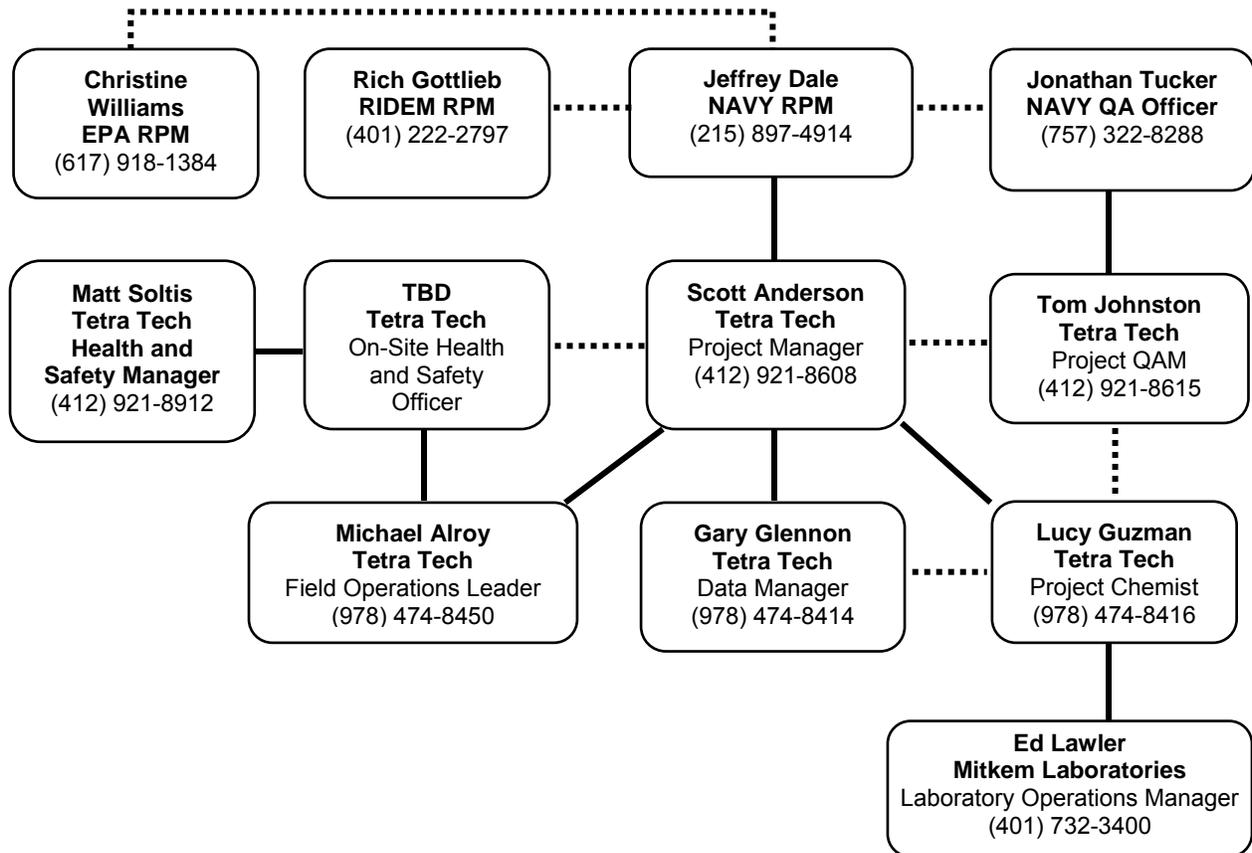
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Stephen Vetere	Tetra Tech, Engineer	(978) 474-8444			
Scott Anderson	Tetra Tech, PM	(412) 921-8608			
Lee Ann Sinagoga	Tetra Tech, Risk Assessment	(412) 921-8887			
Michael Alroy	Tetra Tech FOL, Site Safety Officer	(978) 474-8450			
Tom Johnston	Tetra Tech, Project QAM	(412) 921-8615			
Lucy Guzman	Tetra Tech, Project Chemist	(978) 474-8416			
Ed Lawler	Mitkem Laboratories	(401) 732-3400			

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 Affiliation	Name	Phone Number and/or e-mail	Procedure
Field task modification requests	Tetra Tech FOL	Michael Alroy	(978) 474-8450	FOL gets approval from Tetra Tech PM the same day if possible, or else within one business day. Document via FTMR form.
SAP amendments	Navy RPM	Jeffrey Dale	(215) 897-4914	RPM sends Modification Request to Tetra Tech Program Office within 30 days to initiate changes in scope.
Changes in schedule	Tetra Tech PM	Scott Anderson	(412) 921-8608	PM informs Navy via schedule concurrence letter within one week.
Issues in the field that result in changes in scope of field work	Tetra Tech FOL Tetra Tech PM	Michael Alroy Scott Anderson	(978) 474-8450 (412) 921-8608	FOL informs PM within one business day; PM informs Navy RPM within one business day; RPM issues scope change if warranted within 30 days; scope change to be implemented before work is executed. Document change request on a FTMR form.
Recommendations to stop work and initiate work upon corrective action	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Tetra Tech Site Safety Officer Navy RPM	Michael Alroy Scott Anderson Tom Johnston TBD Jeffrey Dale	(978) 474-8450 (412) 921-8608 (412) 921-8615 TBD (215) 897-4914	Responsible Party informs subcontractors, the Navy, and Project Team within one business day.
Analytical data quality issues	Mitekem Laboratories Tetra Tech Project Chemist Tetra Tech QAM	Ed Lawler Lucy Guzman Tom Johnston	(401) 732-3400 x315 (978) 474-8416 (412) 921-8615	Laboratory notifies Tetra Tech Project Chemist within one business day. Tetra Tech Project Chemist notifies Data Validation Staff and Tetra Tech PM if necessary within one business day.

FTMR - Field Task Modification Request

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

Name	Title/Role	Organizational Affiliation	Responsibilities
Jeffrey Dale	RPM	Navy	Oversee project implementation, including scoping, data review, and evaluation.
Christine Williams	RPM	EPA	Participate in scoping, data review, evaluation, and review of the SAP. Oversee project execution for EPA
Rich Gottlieb	RPM	RIDEM	Participate in scoping, data review, evaluation, and review of the SAP. Oversee project execution for RIDEM.
Scott Anderson	Project Manager	Tetra Tech	Coordinate field work and reporting activities, including sampling, analysis, database management, results reporting, GIS, statistical analyses, and optimization.
Michael Alroy	Field Operations Leader	Tetra Tech	Assist with technical management of the project. Supervise, coordinate, and perform field sampling activities. Prepare data reports.
Tom Johnston	Quality Assurance Manager	Tetra Tech	Ensure quality aspects of the CLEAN program are implemented.
Matt Soltis	Health and Safety Manager	Tetra Tech	Oversee Tetra Tech CLEAN Program safety.
Lucy Guzman	Project Chemist	Tetra Tech	Oversee preparation of chemistry portions of SAP and laboratory scope. Coordinate with laboratory. Oversee performance of Tetra Tech data validation.
Ed Lawler	Laboratory Operations Manager	Mitekem Laboratories	Coordinate analyses with lab chemists, ensure the scope is followed, prepare QA data packages, communicate with Tetra Tech staff.
TBD (Tetra Tech PM will assign)	Field crew members	Tetra Tech	Collect, package, and ship samples in accordance with SAP. Complete field sampling forms.
Gary Glennon	Data Managers, GIS Specialists	Tetra Tech	Consolidate data in database, map data in GIS or other system.

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
Project Name: Confirmatory Sampling and Drain Line Investigation
Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
Document No.: W5209575DF
Revision Number: 0
Revision Date: August 2010

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 will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the accompanying site-specific Tetra Tech Health and Safety Plan (HASP), prepared under separate cover.

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

Project Name: Confirmatory Sampling and Drain Line Investigation Site Name: QDC Outfall 001 Site Location: Former NCBC Davisville, North Kingstown, Rhode Island Projected Date(s) of Sampling: 2010 Project Manager: Stephen Vetere					
Date of Session: January 21, 2009 Scoping Session Purpose: Document characterization and disposal of petroleum-impacted soils and make recommendations for further action. RIDEM and EPA provided comments on this document.					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Curtis Frye	RPM	NAVFAC	(215) 897-4914	Curtis.Frye@navy.mil	Navy project management
David Barney	BEC	NAVFAC	(617) 753-4656	David.A.Barney@navy.mil	Navy management
Stephen Vetere	Project Manager/ Engineer	Tetra Tech	(978) 474-8444	Stephen.Vetere@tetrattech.com	Project management
Richard Gottlieb	RPM	RIDEM	(401) 222-2797 x7138	Richard.Gottlieb@dem.ri.gov	RIDEM project management
Christine Williams	RPM	US EPA Region 1	(617) 918-1384	Williams.Christine@epa.gov	EPA project management

Comments/Decisions:

On behalf of the Navy, Tetra Tech prepared a letter report documenting field activities conducted during the winter of 2008/2009. These activities included characterizing soils that had been stockpiled adjacent to the outfall by the Quonset Development Corporation, and transporting these soils to a non-hazardous waste landfill for disposal. The letter report recommended additional investigations in the outfall area including a) sampling and characterization of the residual material present in the drain pipe and b) a camera survey to evaluate the condition of the pipe and determine its extent.

On February 9, 2009 RIDEM provided comments on the draft letter report. RIDEM concurred with the recommendations made in the document and recommended the collection of samples from the soils downgradient from the outfall. RIDEM also requested the Navy to provide plans depicting the location of the drain line and appurtenances and to excavate test pits along the length of the line to determine if there are impacted soils located along its length. In an email sent on February 9, 2009 EPA agreed with RIDEM's recommendations and requested the Navy to compare soil sampling results to ecological risk-based screening values since the area downgradient from the outfall is a wetland.

Action Item:

Navy will prepare a work plan/SAP for EPA/RIDEM review to support additional investigations at the outfall.

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

Project Name: Confirmatory Sampling and Drain Line Investigation Site Name: QDC Outfall 001 Site Location: Former NCBC Davisville, North Kingstown, Rhode Island Projected Date(s) of Sampling: 2010 Project Manager: Stephen Vetere					
Date of Session: March 12, 2009 Scoping Session Purpose: Discuss scope of work for Outfall 001 investigation.					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Curtis Frye	RPM	NAVFAC	(215) 897-4914	Curtis.Frye@navy.mil	Navy project management
Stephen Vetere	Project Manager/ Engineer	Tetra Tech	(978) 474-8444	Stephen.Vetere@tetrattech.com	Project management

Comments/Decisions:

Curt Frye and Steve Vetere discussed the scope of work to be included in the work plan for additional investigation at Outfall 001. Suggestions provided by EPA and RIDEM will be included in the work plan, except that direct push technology soil borings would be used to investigate soils along the length of the drain pipe rather than test pits.

Action Item:

Navy will prepare a work plan/SAP for EPA and RIDEM review to support additional investigations at the outfall.

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

Project Name: Confirmatory Sampling and Drain Line Investigation Site Name: QDC Outfall 001 Site Location: Former NCBC Davisville, North Kingstown, Rhode Island Projected Date(s) of Sampling: 2010 Project Manager: Scott Anderson					
Date of Session: August – October 2009 Scoping Session Purpose: Comments and Responses on Draft SAP for Confirmatory Sampling and Drain Line Investigation at QDC Outfall 001					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Curtis Frye	RPM	NAVFAC	(215) 897-4914	Curtis.Frye@navy.mil	Navy project management
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Comments/Decisions:

On behalf of the Navy, Tetra Tech prepared a Draft Sampling and Analysis Plan for Confirmatory Sampling and Drain Line Investigation at QDC Outfall 001 dated August 2009

On August 29, 2009 RIDEM provided comments on the Draft SAP and on September 8, 2009 EPA provided comments on the document. EPA and RIDEM recommended the excavation of test pits along the length of the drain line instead of advancement of soil borings. EPA also recommended that VOC and SVOC analyses be added to the target analyte list. The Navy agreed to excavate test pits and to analyze samples collected during the investigation for VOCs and SVOCs.

On October 20, 2009 EPA provided additional comments regarding the Navy's responses. EPA requested the collection of sediment samples from the wetland area located downgradient from the outfall. Navy agreed to collect five sediment samples from the wetland area.

Action Item:

Navy will edit the Draft SAP for EPA and RIDEM review to support additional investigations at the outfall.

SAP Worksheet #10 – Problem Definition
(UFP-QAPP Manual Section 2.5.2)

10.1 SITE LOCATION AND BACKGROUND

The Former NCBC Davisville is located in the town of North Kingstown, Rhode Island and is comprised of three areas: the Main Center (Zones 1 through 4); the West Davisville storage area; and Camp Fogarty, a training facility located approximately 4 miles west of the Main Center (Figure 10-1). A significant portion of the Main Center is situated adjacent to Narragansett Bay. Adjoining the Main Center's southern boundary is the decommissioned Naval Air Station Quonset Point, which was transferred by the Navy to the General Services Administration who in turn transferred portions of the property, between 1975 and 1980, to the Rhode Island Port Authority (now known as Quonset Development Corporation), Town of North Kingstown, and the State of Rhode Island (RIDEM, 2009).

The Former NCBC Davisville was primarily used for training Navy Construction Battalion "Seabees" in construction operations, and as storage and freight yards for construction materials. As a result, the NCBC facility comprised primarily warehouse space and freight yards, most of which are currently demolished, redeveloped, or empty. The Former NCBC Davisville closed on April 1, 1994.

Quonset Development Corporation (QDC) Outfall 001 is located in the central portion of the Former NCBC Davisville facility, to the west of Allen Harbor (Figure 10-1). Installation Restoration (IR) Site 09 (Allen Harbor Landfill) is located to the northeast of the outfall, IR Site 16 (Former Fire Training Area and Creosote Dip Tank) is located to the east and south of the outfall, and the Former Construction Equipment Department ("CED Area") is located to the west. QDC Outfall 001 is located near the intersection of Marine Road and Sanford Road, behind a chain-link fence that surrounds the parking area for recreational users of Allen Harbor Landfill and Calf Pasture Point (Figure 10-2). Undeveloped wetlands are present to the east of the outfall. Groundwater flow direction in the outfall area is toward the east.

Based on a review of historical as-built drawings of drainage systems at the former NCBC, Outfall 001 is the discharge point for an underground drainage line that originated from the former Building 224. Building 224 was part of the CED Area located approximately 1,000 feet to the southwest of the outfall (Figure 10-3). The former Building 224 is the presumed source of contamination present in the outfall area. Building 224 was used by the Navy as a vehicle maintenance and truck washing facility. Contaminated materials from these activities or other historical activities at the CED Area may have been disposed of into the Building 224 drainage system and discharged into the outfall area.

The Navy has conducted the following removal actions at the CED Area:

- At Study Area 01 (Former Drum Storage Area), located approximately 200 feet north of the former Building 224, 55-gallon drums of liquid waste reportedly consisting of waste oil and solvent were removed in 1974.
- At Site 02 (Former Battery Acid Disposal Area), a dry well and leaching field located at the southwest corner of Building 224 were removed in 1996. Excavation of lead-impacted soils was also included in the removal.
- At Study Area 04 (Former Asphalt Disposal Area), located approximately 500 feet west of the former Building 224, asphalt-type material that had been impacted by total petroleum hydrocarbons (TPH) and polychlorinated biphenyls (PCBs) was excavated and removed in 1996.

The CED Area also includes Site 03 (Former Solvent Disposal Area). Between 2001 and 2007, the Navy conducted annual groundwater monitoring of the deep and bedrock aquifers at Site 03 to evaluate concentrations of chlorinated volatile organic compounds (CVOCs) where solvents are reported to have been disposed during Navy operations at the CED Area. The majority of the CVOCs in groundwater at the CED Area, however, are believed to be present as a result of a release from an upgradient property, the PR-58 Nike Site.

Sampling data collected from the CED Area suggest that CVOCs are not present in shallow groundwater in significant concentrations and also that the extent of CVOCs in the deep and bedrock aquifers does not extend to the drain line outfall area (MW-Z3-02, MW01-13D, and MW01-15D/R).

The contaminated materials removed during the removal actions were probably generated from activities at the CED Area, and the same types of materials may have entered into the Building 224 drainage line. Therefore, waste oil and solvents, battery acid lead, TPH, and PCBs that may have entered into the drainage line are the potential sources of contamination present in the outfall area, and the contamination present at QDC Outfall 001 is being addressed under the Navy's IR Program as part of the CED Area.

10.2 PREVIOUS INVESTIGATIONS AND CONCEPTUAL SITE MODEL

During QDC's storm water outfall maintenance activities in the summer of 2008, QDC excavated soil that was present downstream from the drain pipe outlet. During this excavation, QDC observed stained soils and olfactory evidence of contamination. QDC stockpiled this soil adjacent to the outfall (Figure 10-3) and contacted the Navy. Photographs of the outfall area and the drain pipe are provided in Appendix A.

At the BRAC Cleanup Team (BCT) meeting on September 25, 2008, the Navy agreed to characterize and dispose of the soils that were stockpiled adjacent to the outfall. Tetra Tech was contracted by the Navy to

characterize and dispose of the soil stockpile. Tetra Tech awarded a subcontract to Global Remediation Services, Inc. (Global) of Boston, Massachusetts to provide soil characterization, containerization, transportation, and off-site disposal services for the project.

On December 16, 2008, Global collected a composite sample from the QDC Outfall 001 soil stockpile and submitted it to Rhode Island Analytical Laboratories, Inc. of Warwick, Rhode Island for chemical analysis. Based on former site operations at the Construction Equipment Department, the potential soil stockpile contaminants included petroleum products, lead, and polychlorinated biphenyls (PCBs). The soil stockpile sample was analyzed for volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), PCBs, TPH, Resource Conservation and Recovery Act (RCRA) 8 metals, Toxicity Characteristic Leaching Procedure (TCLP) metals, and flashpoint.

Analytical results from the soil stockpile sample indicated the presence of TPH (>10,000 mg/kg), VOCs (primarily methyl-, chloro-, and propyl-benzenes), PCBs (0.3 mg/kg Aroclor-1260), PAHs, and metals (420 mg/kg lead). A copy of the analytical data is provided in Appendix B. The chemical analytes that exceed the Rhode Island Department of Environmental Management (RIDEM) Residential Method 1 Soil Objectives residential Direct Exposure Criteria (DEC) and TPH DEC (RIDEM, 2004), the Residential Soil Regional Screening Levels (RSLs) (December 2009), and/or appropriate selected ecological screening values are presented below in Table 10-1. The ecological screening values presented in the table are the lowest value selected from a group of applicable ecological screening criteria. Although sediments are not present in the excavated area, the area is adjacent to wetlands. The sediment ecological screening values are presented in the table as conservative screening values for soils that may ultimately migrate into a wetland area.

TABLE 10-1

SOIL STOCKPILE SAMPLE ANALYTICAL RESULTS COMPARED TO RIDEM SOIL RESIDENTIAL DEC'S, EPA RESIDENTIAL SOIL RSLs, AND/OR SELECTED ECOLOGICAL SOIL OR SEDIMENT SCREENING LEVELS

Chemical	Stockpile Sample Analytical Result (mg/kg)	RIDEM Method 1 Soil Residential DEC (RIDEM, 2004) (mg/kg)	EPA Residential Soil RSLs (May 2010) ⁽¹⁾ (mg/kg)	Selected Ecological Soil Screening Level ⁽²⁾ (mg/kg)	Selected Freshwater Ecological Sediment Screening Level ⁽³⁾ (mg/kg)
Total Petroleum Hydrocarbons					
C ₁₀ -C ₂₈	10000	500 ⁽⁴⁾	NA	NA	NA
C ₂₈ -C ₃₆	1600	500 ⁽⁴⁾	NA	NA	NA
Volatile Organic Compounds					
Chlorobenzene	2.5	210	29	13.1	0.000842
1,2,4-Trichlorobenzene	0.18	96	6.2	11.1	2.1
1,2,4-Trimethylbenzene	1.2	NA	6.2	NA	NA
1,3,5-Trimethylbenzene	2.0	NA	78	NA	NA
1,2-Dichlorobenzene	0.18	510	190	2.96	0.0165
1,3-Dichlorobenzene	0.14	430	NA	37.7	4.43

Chemical	Stockpile Sample Analytical Result (mg/kg)	RIDEM Method 1 Soil Residential DEC (RIDEM, 2004) (mg/kg)	EPA Residential Soil RSLs (May 2010) ⁽¹⁾ (mg/kg)	Selected Ecological Soil Screening Level ⁽²⁾ (mg/kg)	Selected Freshwater Ecological Sediment Screening Level ⁽³⁾ (mg/kg)
1,4-Dichlorobenzene	0.95	27	2.4	0.546	0.599
Xylenes (total)	0.15	110	63	10	0.16 ⁽⁵⁾
Polycyclic Aromatic Hydrocarbons					
2-Methylnaphthalene	8.3	123	31	29 ⁽⁶⁾	0.0202
Acenaphthene	6.3	43	340	20 ⁽⁷⁾	0.0067
Anthracene	8.6	35	1700	29 ⁽⁶⁾	0.0572
Benzo(a)anthracene	17	0.9	0.15	1.1 ⁽⁶⁾	0.108
Benzo(a)pyrene	16	0.4	0.015	1.1 ⁽⁶⁾	0.15
Benzo(b)fluoranthene	26	0.9	0.15	1.1 ⁽⁶⁾	0.13 ⁽⁸⁾
Benzo(g,h,i)perylene	5.4	0.8	170 ⁽⁹⁾	1.1 ⁽⁶⁾	0.17
Benzo(k)fluoranthene	20	0.9	1.5	1.1 ⁽⁶⁾	0.24
Chrysene	20	0.4	15	1.1 ⁽⁶⁾	0.166
Dibenzo(a,h)anthracene	3.1	0.4	0.015	1.1 ⁽⁶⁾	0.033
Fluoranthene	48	20	230	29 ⁽⁶⁾	0.423
Fluorene	9.0	28	230	1.1 ⁽⁶⁾	0.0774
Indeno(1,2,3-c,d)pyrene	6.9	0.9	0.15	1.1 ⁽⁶⁾	0.017
Phenanthrene	24	40	170 ⁽⁹⁾	1.1 ⁽⁶⁾	0.204
Pyrene	32	13	170	1.1 ⁽⁶⁾	0.195
Polychlorinated Biphenyls					
Aroclor-1016	0.2	10 ⁽¹⁰⁾	0.39	0.000332	0.0598 ⁽¹⁰⁾
Aroclor-1260	0.3	10 ⁽¹⁰⁾	0.22	0.000332	0.0598 ⁽¹⁰⁾
Metals					
Arsenic	4.5	7	0.39	18 ⁽⁶⁾	9.8
Barium	100	5500	1500	330 ⁽⁶⁾	48 ⁽⁸⁾
Cadmium	3.0	39	7	0.36 ⁽⁶⁾	0.99
Chromium	48	390	0.29 ⁽¹¹⁾	0.4 ⁽¹²⁾	43.4
Lead	420	150	400	11 ⁽⁶⁾	35.8
Mercury	0.17	23	2.3	0.1 ⁽¹²⁾	0.18

Exceeded criteria shown in **bold** font.

- EPA Regions 3, 6, 9 Regional Screening Levels (RSLs), Residential Soil value (USEPA, 2010). 1/10 RSL is presented for non-carcinogenic analytes to correspond to a Hazard Quotient = 0.1. If the non-carcinogenic 1/10 RSL value is less than the carcinogenic value for an analyte, the non-carcinogenic 1/10 RSL value is presented.
- Screening level is the Region 5 Ecological Screening Level (USEPA, 2003), unless otherwise noted.
- Screening level is the Region 3 BTAG Freshwater Sediment Screening Benchmark (USEPA, 2006), unless otherwise noted.
- Value is the residential TPH DEC.
- Value is the Secondary Chronic Value (SCV) (Jones, et al., 1997).
- Value is EPA Soil Screening Level (USEPA, 2003, 2005, 2006, 2007, 2008).
- Value is ORNL Plant screening value (Efroymson, et al, 1997a).
- NOAA SQuiRT Sediment Benchmark (Buchman, 2008). Value is the saltwater benchmark.
- Value is for pyrene.
- Value is for total PCBs.
- Value is for hexavalent chromium.
- Value is ORNL Invertebrate screening value (Efroymson, et al, 1997b).

BTAG = Biological Technical Assistance Group

DEC = Direct Exposure Criteria

EPA = U.S. Environmental Protection Agency

ORNL = Oak Ridge National Laboratory

NA = Not available

NOAA SQuiRT = National Oceanic and Atmospheric Administration Screening Quick Reference Tables

RSL – Regional Screening Level

RIDEM – Rhode Island Department of Environmental Management

TPH – Total Petroleum Hydrocarbons

As shown in the table, TPH exceeds the RIDEM TPH DEC; select PAHs and metals exceed some or all of the criteria; and select PCBs exceed the RSL and ecological screening values. No VOCs exceed the residential DEC or RSLs, and only a few VOCs exceed the soil or sediment ecological values.

On December 29, 2008 Global mobilized an excavator to the site and removed the soil stockpile from the Outfall 001 area, loading the soil into two roll-off containers. The containers were transported to the Tetra Tech project area and staged behind a locked gate. Global transported the soil roll-off containers (one containing 10.37 tons of soil and the other, 12.54 tons) under a non-hazardous waste manifest to ESMI of New Hampshire.

It is the project team's assumption that the source of contamination to the soils downstream of the drain pipe outlet in the excavation area was the discharge from the drainage pipe that formerly originated from Building 224. The drainage pipe has been inactive since at least 2006 when Building 224 was demolished. Chemicals used during historical truck maintenance activities (and other historical activities that occurred in and around the Building 224 area) probably entered the drainage system and were transported along the length of the pipe, discharging into soils in the drainage swale downstream from the pipe and potentially migrating with the flow of surface water further downstream toward the wetland area. Contaminants migrating toward the wetland area will either accumulate in the wetland or be transported into Allen Harbor.

In addition to the potential for contamination in the outfall area soil and sediment, there is a potential for contamination in subsurface soil adjacent to the underground drain pipe. Given the age of the drain pipe, it is reasonable to expect that it may have been compromised along its length and some contaminated materials may have leaked into the soils adjacent to the pipe, thereby potentially contaminating nearby subsurface soils and downgradient groundwater.

Access to the outfall area is currently restricted by the chain link fence that surrounds the parking area that is located along Sanford Road. Persons currently accessing the site include trespassers, although there are recreational areas in close proximity to the outfall. Given that the future land use is unknown, it is customary to evaluate use of a property as residential and recreational. Therefore, potential future human receptors include residents and recreational users, in addition to industrial and construction workers and trespassers. Ecological receptors include animal and plant species that could be affected by the contaminants present in environmental media. Human and ecological receptors may be affected by contaminants in the excavation area, and human receptors may be affected by subsurface soil in the area alongside the drain line. Ecological receptors typically are not exposed to subsurface media (greater than 3 feet bgs).

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

The following text describes the development of project quality objectives (PQOs) using EPA's data quality objective (DQO, Systematic Planning) process.

11.1 PROBLEM STATEMENTS

As discussed above, the historical use of oil and hazardous materials at the CED may have resulted in the release of contaminants into the drainage system of Building 224 where they migrated toward the east through a drainage pipe which ultimately discharged at QDC Outfall 001. Consequently, the following problem statements need to be resolved:

Problem Statement 1: The project team must determine whether or not there are contaminants in soils remaining in the excavation area downstream from the outfall, and in sediments in the wetland area located downgradient from the outfall, at concentrations that might present a risk to human health or the environment.

Problem Statement 2: The project team must determine whether or not residual material present in the drain pipe is providing a continuing source of contamination to the area downstream from the drain pipe outlet and the wetland area.

Problem Statement 3: The project team must determine whether or not there are areas of compromised integrity along the length of the pipe and, if so, whether contaminants have been released to the subsurface soil adjacent to the pipe at concentrations that might present a risk to human health.

11.2 INPUTS TO PROBLEM RESOLUTION

Data and information that will be required to resolve the problems identified in Section 11.1 include a drainage pipe reconnaissance, chemical analytical data (soil and sediment), and decision criteria for making the determinations to resolve the problems.

11.2.1 Drainage Pipe Reconnaissance

A pipe reconnaissance will be conducted using remote video technology to evaluate the condition of the pipe along its entire length. The reconnaissance will be used to document the condition of the pipe and identify areas of compromised integrity.

11.2.2 Chemical Analytical Data

The concentrations of potential contaminants in soil and sediment must be quantified in order to address the Problem Statements. The concentrations of VOCs, SVOCs (including PAHs), GRO (C₅ – C₁₂), Extractable TPH (ExTPH) (C₉ – C₄₀), PCBs, and TAL metals in soil, sediment, and residual material are needed for decision making. The list of target chemical analytes for soil, sediment, and residual material samples is presented in Worksheet #15.

11.2.3 Project Screening Levels

The Project Screening Levels (PSLs) associated with the target analytes for soil (confirmatory soil and test pit soil) and wetland sediment are presented in Worksheet #15. Also presented in Worksheet #15 are the fixed laboratory analytical methods needed for the laboratory to achieve LOQs and LODs less than or equal to the PSLs.

The PSLs for confirmatory soil samples will be the lowest of the screening criteria presented below:

- EPA Regions 3, 6, and 9 Regional Screening Values (RSLs) for Chemical Contaminants at Superfund Sites, Residential Soil Values (May 2010) (USEPA, 2010).
- RIDEM Residential DEC for TPH in soil (RIDEM, 2004)
- The appropriate ecological soil screening levels selected from among:
 - EPA Ecological Soil Screening Levels for plants, invertebrates, and wildlife (USEPA, 2003, 2005, 2006, 2007, 2008) ORNL Toxicological Benchmarks for plants (Efroymsen, et al, 1997a)
 - ORNL Toxicological Benchmarks for invertebrates (Efroymsen, et al, 1997b)
 - EPA Region 5 Ecological Screening Screening Levels (USEPA, 2003).

The PSLs for test pit soil will be the EPA Regions 3, 6, and 9 RSLs, Residential Soil Values. Ecological screening criteria are not applicable because soil sampling will be at least 3 feet bgs.

The PSLs for sediment will be the appropriate freshwater ecological sediment screening levels selected from among:

- USEPA Region 3 Biological Technical Assistance Group (BTAG) Freshwater Sediment Screening Benchmarks (USEPA, 2006).

- Sediment screening values derived using equilibrium partitioning theory such as the Secondary Chronic Values (SCV) (Table 3 in Jones, et al., 1997) and the Ecotox Thresholds Sediment Quality Benchmarks (SQBs) (USEPA, 1996)
- National Oceanographic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQuiRTs) sediment benchmarks (Buchman, M. F., 2008)

Project screening levels are not applicable for residual material for this project because residual material will not be screened against fixed criteria. Instead, residual material chemical concentrations will be compared with the concentrations in downgradient soil and sediment (see Section 11.4.B), and those concentrations are not yet known. However, for the purpose of determining the analytical methods and laboratory LOQs and LODs needed to allow comparison of residual material concentrations with detected levels of soil and sediment, values equal to the lower of the PSLs for confirmatory soil and wetland sediment are presented for residual material in WS 15c.

11.3 DEFINE THE STUDY BOUNDARY

The populations of interest are soil and sediment downstream/downgradient from the QDC Outfall 001; residual material present within the drain pipe; and soil located adjacent to the drain pipe. These media potentially contain contaminants exceeding the PSLs (or other screening criteria) due to the release of contaminants into the drainage system of the former Building 224.

The depth interval of interest for soil and sediment samples collected downstream from the outfall is 0- to 12-inches bgs. Data will be collected from the footprint of the 2008 soil excavation area and from within the wetland area located downgradient from the excavation area.

Residual material concentration data will be collected inside the drain pipe, within two feet of the outfall outlet, and in areas of compromised portions of the pipe, should residual materials be encountered during investigative activities.

Subsurface soil data will be collected from areas adjacent to the drain line, which extends approximately 1,200 feet from the former Building 224 to QDC Outfall 001. The depth/location of the pipe will be identified by test pitting to ensure soil samples are collected from an appropriate depth. It is assumed that the pipe is located approximately 3 feet bgs, therefore subsurface soil concentrations will be quantified from the 3- to 5-foot depth interval.

The pipeline reconnaissance will be conducted prior to data collection adjacent to the pipe since the location of investigations will be contingent upon the findings of the reconnaissance. Vegetation clearing

and clearing of obstructions may be necessary prior to these activities to facilitate the pipeline reconnaissance and subsurface investigation, respectively.

The samples proposed in this SAP will be collected during a single sampling event. If decisions are made to conduct additional sampling to further characterize the nature and extent of contamination, an addendum to the SAP will be prepared to support another sampling event.

11.4 ANALYTIC APPROACH

A series of decision rules was developed to govern data use and decision making. The decision rules are presented in this section.

- A. Evaluate potential risks associated with contamination in soil and sediment downgradient from QDC Outfall 001.
 1. Compare the concentrations of contaminants in soil and sediment with the PSLs. If no criteria are exceeded, conclude that all of the contaminated soil present in the outfall area was removed, contaminants have not migrated into the wetland area, and no further sampling is needed in these areas.
 2. If any of the PSLs in any soil or sediment sample is exceeded, decide whether additional sampling is needed to further characterize the nature and extent of contamination:
 - Evaluate the exceedances in terms of their magnitude, number, and location (spatial distribution). If the number of chemicals with exceedances is relatively few, the magnitudes of exceedances are relatively small, exceedances of probable effects concentrations (PECs) are relatively few (see next bullet), and exceedances are spatially scattered, the BCT may decide that further sampling is not needed.
 - Consider the conservative nature of the TECs as ecological sediment screening criteria. The PEC is a less conservative threshold than the TEC. For each exceedance of a TEC, compare the sample result with the PEC. Results between the TEC and the PEC may be considered a “gray area” in terms of degree of risk to ecological receptors.
 3. If additional investigation beyond the scope of this SAP is determined to be necessary based on the data collected under this SAP, an addendum will be prepared (if the additional work is needed to achieve the objectives of this SAP) or a separate SAP will be prepared (if the additional investigations are proposed to achieve a new set of objectives).

B. Evaluate whether residual material in pipe provides a continuing source of contamination to the outfall area and downgradient wetland.

1. Evaluate each of the following lines of evidence:

- The similarity between contaminants present in the residual material and those in downgradient soil and sediment.
- The spatial distribution and concentrations of contaminants in soil and sediment.
- The absolute concentrations of the contaminants in the residual material, and the concentrations relative to those detected in soil and sediment.

2. Use a weight-of-evidence approach to assess the likelihood that the residual material in the pipe, or in areas of compromised pipe, was, and may continue to be, a source of contamination to soils in the excavation area and sediments in the wetland area. In general, if chemicals detected in the confirmatory samples or wetland sediment samples largely match those detected in the residual material, the likelihood is high that the material within the pipe was a source of contamination to the soil in the outfall area and sediments in the wetland area. If the concentrations of chemicals detected in the residual material are high, the likelihood is high that the material within the pipe is a continuing source of contamination. If the concentrations are low, the likelihood is more uncertain.

- The relative concentrations in the residual material sample and the confirmatory samples and wetland sediment samples will be considered, but they will not be definitive in confirming residual material as a source or in estimating the degree to which the continuing source may affect the area; contaminant concentrations might be expected to decrease as chemicals migrate away from the source, but concentrations in the affected area may increase as chemicals accumulate over time.
- If the residual material in the drain pipe or in areas of compromised pipe is likely a continuing source of contamination to the soils in the excavation area and sediments in the wetland area, then the BCT will meet to evaluate the need for remediation of the drain pipe and develop the next appropriate steps. Remediation of the potential source would be required prior to taking remedial action to address the soils, in the excavation area, and/or sediments in the wetlands downstream from the pipe outlet. If the residual material is not acting as a continuing source of contamination, no remediation of the drain pipe is required with respect to Decision 2.

- C. Evaluate potential risks associated with contamination in subsurface soil adjacent to the drainage pipe by identifying potentially compromised sections of the underground drain line and collecting soil concentration data from the depth interval immediately below the pipe elevation.
1. Compare subsurface soil concentrations to PSLs. If no criteria are exceeded, no further sampling in this area is needed.
 2. If the PSL is exceeded at any location, evaluate the exceedances in terms of the magnitude and number. If the number of chemicals with exceedances is relatively few and the magnitude of exceedances is relatively small, the BCT may decide that further sampling is not needed.
 3. If additional investigation beyond the scope of this SAP is determined to be necessary based on the data collected under this SAP, an addendum will be prepared (if the additional work is needed to achieve the objectives of this SAP) or a separate SAP will be prepared (if the additional investigations are proposed to achieve a new set of objectives). Decisions regarding the need for additional characterization will be made in collaboration with the BCT.

Decisions regarding the need for additional characterization will be made in collaboration with the BCT.

11.5 SPECIFY PERFORMANCE CRITERIA

The sample locations were selected based on the need to characterize the presence and nature, and preliminary extent, of contamination associated with releases to the drainage system in the former Building 224 that have migrated toward QDC Outfall 001. The soil and sediment analytical data will be used to map the spatial boundaries of environmental media containing contaminant concentrations exceeding PSLs. Because sample locations depend on biased (non-random) sampling, probability limits for false positive and false negative decision errors were not established for this project. Simple comparisons of measured chemical concentrations to PSLs will be made and the project team will use the results to determine whether the amount and type of data collected are sufficient to evaluate potential risks associated with contamination detected. Particular scrutiny will be applied to analytical results below the LOQ when PSLs are below the LOQ. The data usability evaluation process is described in more detail in Worksheet #37.

11.6 DEVELOP THE DATA COLLECTION PLAN

The sampling plan and rationale are presented in Worksheet #17.

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
 Project Name: Confirmatory Sampling and Drain Line Investigation
 Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

SAP Worksheet #12 – Measurement Performance Criteria Table (note matrix in table entry)

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Temperature Blank	All analytical groups	One per cooler	Accuracy/bias/representativeness	4° C ± 2° C	S
Trip Blank	VOCs, GRO	One per cooler	Bias/contamination	No target analytes > ½ LOQ (>LOQ for common laboratory contaminants), unless target analytes in field samples are > 10x those in trip blank.	S & A
Field Duplicates	VOCs, SVOCs, GRO, DRO, PAHs, PCBs	One per 10 samples	Precision/comparability	Soils: RPD ≤ 50%; Waters: RPD ≤ 30%. If samples results are < 2x LOQ, professional judgment is used.	S & A
	Metals	One per 10 samples	Precision/comparability	Values ≥ 5x LOQ: RPD ≤ 50% for soils; RPD ≤ 30% for waters. Values < 5x LOQ: Absolute Difference ≤ 4x LOQ for soils; Absolute Difference ≤ 2x LOQ for waters.	S & A
All samples	All analytical groups	All samples	Sensitivity	LOQ < project action limits listed in Worksheet #15	A
All samples	All analytical groups	All samples	Data completeness	95% overall	S & A

Note: The measurement performance criteria for laboratory QC samples are presented in Worksheet #28.

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
Project Name: Confirmatory Sampling and Drain Line Investigation
Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
Document No.: W5209575DF
Revision Number: 0
Revision Date: August 2010

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
Characterization of stockpile of soil excavated from area downstream from outfall	Tetra Tech; Final Outfall 001 Soil Characterization and Removal Completion Report	One composite sample collected from soil stockpile on 12/16/08. Sample analyzed by RI Analytical Laboratories, Inc., for VOCs, PAHs, PCBs, TPH, RCRA 8 metals, TCLP metals, and flashpoint.	Data were used to characterize excavated soils for proper off-site disposal. Data were used to select the analytical groups for soil sampling proposed in this SAP.	None

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

The following project tasks are summarized below:

- Field Tasks
- Analytical Tasks
- Data Management
- Project Report

Field documentation forms to be utilized during the investigation are provided in Appendix C. The SOPs referenced below are included in Appendices D (Tetra Tech SOPs) and E (Laboratory SOPs). Project-specific procedures for test pitting, soil sampling, sediment sampling, and residual material sampling are also provided in Appendix F. The field team will follow the project-specific field procedures unless these procedures do not provide guidance on a specific field task issue. In that case, the procedures in the cited SOPs will be followed.

14.1 FIELD TASKS

Field tasks required to complete the work included in this SAP are described in this section.

14.1.1 Mobilization/Demobilization

Mobilization includes procurement of field equipment and supplies; kick-off meetings and project orientation activities; mobilization of field staff, equipment, and supplies to the site; and site set-up. Mobilization will include an on-site field team orientation and health and safety briefing.

A field team orientation meeting will be conducted prior to starting the fieldwork to familiarize the team personnel with site-specific health and safety requirements, objectives and scope of the field activities, chain of command, and lines of communication. This meeting will be attended by the field staff, project manager (PM), field operations leader (FOL), site safety officer (SSO), lead geologist, and project chemist.

14.1.2 Utility Clearance

A DIGSAFE number will be obtained at all potential test pitting or other subsurface investigation locations prior to sample collection, in accordance with SOP HS-1.0.

14.1.3 Drainage Pipe Reconnaissance

A video inspection of the interior of the drain line will be performed to evaluate the structural integrity of the drain line. A subcontractor specializing in utility pipe inspection will be procured to conduct the reconnaissance. The inspection equipment will be capable of documenting conditions along the entire length of the drain. If the reconnaissance determines that the integrity of the drain line has been compromised, test pitting will be conducted to evaluate the extent of the damage and to enable the collection of soil samples from soils located adjacent to the pipe.

The extension of the inspection equipment may be impeded by residual material or other obstructions in the drain line. If so, measures will be taken, as practical, to clear the line of obstructions.

14.1.4 Soil, Sediment, and Residual Material Sampling

The sampling design is detailed in Worksheet #17. Sample locations are presented in Worksheet #18 and on Figures 17-1 and 17-2.

Confirmatory Soil Sampling. Soil samples will be collected from the excavation area downstream from the drain pipe outlet, where soils were removed during the summer of 2008 (Figure 10-3). Bottom and sidewall samples will be collected according to SOP SA-1.3 and the project-specific sampling procedures presented in Appendix F.

Sediment Sampling. Sediment samples will be collected from the wetland area located downstream from the outfall excavation area. Sediment samples will be collected according to Tetra Tech SOP SA-1.2 and the project-specific sampling procedures presented in Appendix F.

Test Pitting/Soil Sampling Along Drain Line. Test pitting will be completed along the length of the drain pipe (Figure 10-3) according to the decision rules presented on Worksheet #11 and Section 14.1.3.

As-built drawings of the drainage line and pipe reconnaissance information will be utilized to select test pitting locations. Test pits will be excavated using a rubber-tired backhoe to a depth of up to 8 feet. The depth/location of the pipe will be identified by test pitting to ensure soil samples are collected from an appropriate depth (assume 3- to 5-foot depth interval bgs). Soil samples will be collected for laboratory analysis from each test pit. The soil samples will be collected according to SOP SA-1.3 and the project-specific sampling procedures presented in Appendix F. Soil sample collection will be biased toward areas where contamination is observed either through field screening, visual, or olfactory evidence. After samples are collected, test pits will be backfilled with native materials to the original grade.

Residual Material Sampling. Residual material samples will be collected from the interior of the drain pipe to evaluate concentrations of contaminants. The samples will be collected according to the project-specific sampling procedures presented in Appendix F.

14.1.5 Field Quality Control (QC) Samples

Field QC samples will be collected as part of the investigation, including field duplicates and trip blanks. Worksheet #20 presents the field QC sample summary. Also, additional sample volume will be collected as necessary for the laboratory QC analysis of matrix spike (MS) and matrix spike duplicate (MSD) analysis (organics), MS and laboratory duplicate analysis (metals).

14.1.6 Equipment Decontamination

All non-disposable equipment that comes in contact with the sample medium will be decontaminated according to Tetra Tech SOP SA-7.1 and Appendix F to prevent cross-contamination between sampling points. This includes equipment such as the excavator bucket and stainless steel bowls, scoops, as well as other non-disposable equipment used to collect samples for laboratory analysis. Personnel decontamination is discussed in the project-specific Health and Safety Plan (HASP).

14.1.7 Investigation-Derived Waste (IDW) Characterization and Disposal

IDW generated during the investigation will include decontamination fluids, used personal protective equipment (PPE), used sampling equipment, and excess sample material. IDW generated during this field investigation will be containerized into 55-gallon drums or polyethylene tanks and staged at the Navy's field office trailer. IDW characterization and disposal will be performed after all IDW has been containerized. IDW will be managed in accordance with Tetra Tech SOP SA-7.1.

14.2 ANALYTICAL TASKS

Chemical analysis for VOCs, SVOCs, GRO, ExTPH, PAHs, PCBs, and TAL metals will be performed by a subcontracted laboratory, Mitkem Laboratories (Mitkem), of Warwick, Rhode Island.

Analyses will be performed in accordance with the analytical methods identified in Worksheet #19 and with the requirements of the analytical specifications for laboratory services developed by Tetra Tech for this work (Appendix E). The subcontracted laboratory will meet the PQLs specified in Worksheet #15.

The analytical specification details the analytical requirements, number of samples, matrix, analytical method to be performed, preservatives, holding times, the quantitation limits required for the project, and data deliverables. Because the laboratory subcontract was issued prior to completion of this SAP, some provisions of the analytical specification may be modified to meet this SAP. The laboratory will perform the chemical analyses following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheet #19. Copies of the laboratory SOPs (Mitekem Laboratories) are included in Appendix G.

14.3 DATA MANAGEMENT AND REVIEW

Data management and review tasks are described below and in other worksheets in this SAP, as noted below.

Project Documentation and Records

- Field sample collection and field measurement records are described in Worksheets 27 and 29.
- Laboratory data package deliverables are described in the analytical specification (Appendix E).
- Data assessment documents and records are listed in Worksheet #29.

Data Recording Formats are described in Worksheet #27.

Data Handling and Management. After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

Data Tracking and Control. The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking.** Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted laboratory. Upon receipt of the data packages from the analytical laboratory, the Project Chemist will oversee the data validation effort, which includes verifying that

the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

- **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.
- **Data Security.** The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Data Review

- Data verification processes are described in Worksheet #34.
- Data validation processes are described in Worksheets #35 and #36.
- Usability assessment processes are described in Worksheet #37.

Assessment and Oversight

Refer to Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

14.4 PROJECT REPORT

Draft and Final versions of the project report will be prepared and submitted to EPA and RIDEM for review. The format of the project report will be:

- Section 1.0 Introduction
 - Project objectives
 - Site background
 - Report organization

- Section 2.0 Field Investigation Description
 - Confirmatory soil sampling
 - Sediment sampling
 - Residual material sampling
 - Video investigation
 - Test pitting and subsurface soil sampling

- Section 3.0 Field Investigation Results
 - Summary of previous analytical results for soil stockpile sample
 - Confirmatory sample analytical results
 - Sediment sample analytical results
 - Residual material sample analytical results
 - Test pit soil sample analytical and screening results

- Section 4.0 Summary and Conclusions
 - Presence or absence of contaminants above screening values
 - Preliminary nature and extent based on sampling results
 - Recommendations for future actions or no further action

- Appendices

The Navy will develop responses to comments received on the draft reports. The draft and final versions of these reports will be submitted in hardcopy and electronic format to the project stakeholders.

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
 Project Name: Confirmatory Sampling and Drain Line Investigation
 Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

SAP Worksheet #15a – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Notes for all matrices: Sample analytical results for VOCs, SVOCs, PCBs, and Metals will be reported down to the Detection Limit (DL), with non-detected results qualified as “U” and reported with an associated value of the Limit of Detection (LOD); positive results between the LOQ and DL will be qualified as “J” (estimated) due to uncertainty below the LOQ. Sample results for GRO, ExTPH, and PAHs will be reported down to the LOQ. Mitkem does not report GRO and ExTPH results below the LOQ due to lack of confirmation column analysis in the method. PAHs will be analyzed in SIM mode; Mitkem does not report SIM results below the LOQ due to limited mass spectral data, which can lead to false positive detections. For the GRO, ExTPH, and PAH analyses, Mitkem will analyze a standard at the LOQ.

Mitkem will analyze for and report individual Aroclors and Tetra Tech will calculate PCBs (Total).

Select metals (arsenic, cobalt, silver, and thallium) will be analyzed by EPA SW-846 Method 6020A, instead of Method 6010C, in order to achieve lower detection limits to meet the PSLs. Although not all of these metals would require the lower detection limits for each matrix, the same metals will be analyzed by Method 6020A across all matrices in order to simplify the sampling and analysis procedures.

In this Worksheet #15, the Project Screening Level (PSL) is presented in bold font if it is less than the LOQ but greater than or equal to the LOD; and the PSL is presented as bolded and shaded if it is less than the LOD. The limitations on data usability due to unmet sensitivity goals will be evaluated as described in Worksheet #37 and discussed in the project report..

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Volatile Organic Compounds									
1,1,1-Trichloroethane	71-55-6	8260C	870	29.8	Region 5 ESL	9.9	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	0.56	0.127	Region 5 ESL	0.042	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.1	1.1	EPA RSL Res	0.37	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	3.3	3.3	EPA RSL Res	1.1	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	24	8.28	Region 5 ESL	2.8	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	190	2.96	Region 5 ESL	0.99	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.43	0.43	EPA RSL Res	0.14	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	0.89	0.89	EPA RSL Res	0.3	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	6.2	6.2	EPA RSL Res	2.1	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	6.2	6.2	EPA RSL Res	2.1	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	78	78	EPA RSL Res	26	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	--	37.7	Region 5 ESL	13	0.005	0.002	0.0007
1,4 Dichlorobenzene	106-46-7	8260C	2.4	0.546	Region 5 ESL	0.18	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	2800	89.6	Region 5 ESL	30	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	21	12.6	Region 5 ESL	4.2	0.005	0.004	0.00083
4-Methyl-2-Pentanone	108-10-1	8260C	530	443	Region 5 ESL	150	0.005	0.004	0.00073

Project-Specific Sampling and Analysis Plan

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 Project Name: Confirmatory Sampling and Drain Line Investigation
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Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Acetone	67-64-1	8260C	6100	2.5	Region 5 ESL	0.83	0.005	0.004	0.0016
Benzene	71-43-2	8260C	1.1	0.255	Region 5 ESL	0.085	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	0.27	0.27	EPA RSL Res	0.09	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	61	15.9	Region 5 ESL	5.3	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	0.73	0.235	Region 5 ESL	0.078	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	82	0.0941	Region 5 ESL	0.031	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.61	0.61	EPA RSL Res	0.2	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	29	13.1	Region 5 ESL	4.4	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	1500	1500	EPA RSL Res	500	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.29	0.29	EPA RSL Res	0.097	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	12	10.4	Region 5 ESL	3.5	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	78	0.78373	Region 5 ESL	0.26	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	1.7	0.398	Region 5 ESL	0.13	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	0.68	0.68	EPA RSL Res	0.23	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	5.4	5.16	Region 5 ESL	1.7	0.005	0.002	0.0005
Methylene Chloride	75-09-2	8260C	11	4.05	Region 5 ESL	1.4	0.005	0.002	0.0013
Styrene	100-42-5	8260C	630	4.69	Region 5 ESL	1.6	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.55	0.55	EPA RSL Res	0.18	0.005	0.002	0.00062
Toluene	108-88-3	8260C	500	5.45	Region 5 ESL	1.8	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	15	0.784	Region 5 ESL	0.26	0.005	0.002	0.00053
trans-1,3-Dichloropropene	10061-02-6	8260C	1.7	0.398	Region 5 ESL	0.13	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	2.8	2.8	EPA RSL Res	0.93	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	79	16.4	Region 5 ESL	5.5	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	0.06	0.06	EPA RSL Res	0.02	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	63	10	Region 5 ESL	3.3	0.005	0.002	0.00047
Semivolatile Organic Compounds									
2,4,5-Trichlorophenol	95-95-4	8270D	610	4	ORNL Plant	1.3	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	6.1	6.1	EPA RSL Res	2	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	18	18	EPA RSL Res	6	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	120	0.01	Region 5 ESL	0.0033	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	12	0.0609	Region 5 ESL	0.02	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	1.6	1.28	Region 5 ESL	0.43	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	6.1	0.0328	Region 5 ESL	0.011	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	630	0.0122	Region 5 ESL	0.0041	0.33	0.067	0.052

Project-Specific Sampling and Analysis Plan

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Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
2-Chlorophenol	95-57-8	8270D	39	0.243	Region 5 ESL	0.081	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	310	40.4	Region 5 ESL	13	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	61	61	EPA RSL Res	20	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	12	1.6	Region 5 ESL	0.53	0.33	0.067	0.022
3,3'-Dichlorobenzidine	91-94-1	8270D	1.1	0.646	Region 5 ESL	0.22	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	--	3.16	Region 5 ESL	1.1	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	0.49	0.144	Region 5 ESL	0.048	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	--	--	--	--	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	610	7.95	Region 5 ESL	2.7	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	2.4	1.1	Region 5 ESL	0.37	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	31	31	EPA RSL Res	10	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	24	21.9	Region 5 ESL	7.3	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	--	5.12	Region 5 ESL	1.7	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	18	0.302	Region 5 ESL	0.1	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	0.21	0.21	EPA RSL Res	0.07	0.33	0.167	0.074
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	35	0.925	Region 5 ESL	0.31	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	260	0.239	Region 5 ESL	0.08	0.33	0.067	0.0063
Carbazole	86-74-8	8270D	--	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	7.8	7.8	EPA RSL Res	2.6	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	4900	24.8	Region 5 ESL	8.3	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	4900	734	Region 5 ESL	240	0.33	0.067	0.0065
Di-n-butyl phthalate	84-74-2	8270D	610	0.15	Region 5 ESL	0.05	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	--	709	Region 5 ESL	240	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	6.1	0.0398	Region 5 ESL	0.013	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	37	0.755	Region 5 ESL	0.25	0.33	0.067	0.032
Hexachloroethane	67-72-1	8270D	6.1	0.596	Region 5 ESL	0.2	0.33	0.167	0.096
Isophorone	78-59-1	8270D	510	139	Region 5 ESL	46	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	4.8	1.31	Region 5 ESL	0.44	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	0.069	0.069	EPA RSL Res	0.023	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	99	0.545	Region 5 ESL	0.18	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	3	2.1	EPA SSL Wildlife	0.7	0.67	0.167	0.082
Phenol	108-95-2	8270D	1800	30	ORNL Invert	10	0.33	0.067	0.029

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Polycyclic Aromatic Hydrocarbons									
2-Methylnaphthalene	91-57-6	8270D SIM	31	29	EPA SSL Invert	9.7	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	340	20	ORNL Plant	6.7	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	340	29	EPA SSL Invert	9.7	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	1700	29	EPA SSL Invert	9.7	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.015	0.015	EPA RSL Res	0.005	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	170	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	1.5	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	15	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.015	0.015	EPA RSL Res	0.005	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	230	29	EPA SSL Invert	9.7	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	230	29	EPA SSL Invert	9.7	0.0033	0.0033	0.00057
Indeno(1,2,3-c,d)pyrene	193-39-5	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	3.6	3.6	EPA RSL Res	1.2	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	170	29	EPA SSL Invert	9.7	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	170	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.0017
Polychlorinated Biphenyls (PCBs)									
Aroclor-1016	12674-11-2	8082A	0.39	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.14	0.000332	Region 5 ESL	0.00011	0.033	0.0166	0.0038
Aroclor-1232	11141-16-5	8082A	0.14	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.11	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	--	0.000332	Region 5 ESL	0.00011	0.033	0.0166	0.0039
Metals									
Aluminum	7429-90-5	6010C	7700	50	ORNL Plant	17	10	2.5	1.2
Antimony	7440-36-0	6010C	3.1	0.27⁽⁶⁾	EPA SSL Wildlife	0.09	1	0.35	0.18
Arsenic	7440-38-2	6020A	0.39	0.39	EPA RSL Res	0.13	0.5	0.20	0.065
Barium	7440-39-3	6010C	1500	330	EPA SSL Invert	110	10	0.7	0.38

Project-Specific Sampling and Analysis Plan

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 Project Name: Confirmatory Sampling and Drain Line Investigation
 Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Beryllium	7440-41-7	6010C	16	10	ORNL Plant	3.3	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	7	0.36	EPA SSL Wildlife	0.12	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	0.29	0.29⁽⁶⁾	EPA RSL Res	0.097	1	0.1	0.054
Cobalt	7440-48-4	6020A	2.3	2.3	EPA RSL Res	0.77	0.5	0.20	0.089
Copper	7440-50-8	6010C	310	28	EPA SSL Wildlife	9.3	1.5	0.75	0.42
Iron	7439-89-6	6010C	5500	200	ORNL Invert	67	10	5	2.2
Lead	7439-92-1	6010C	400	11	EPA SSL Wildlife	3.7	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	180	180	EPA RSL Res	60	2.5	1	0.47
Mercury	7439-97-6	7471B	2.3	0.1	ORNL Invert	0.033	0.033	0.02	0.002
Nickel	7440-02-0	6010C	150	38	EPA SSL Plant	13	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	39	0.52⁽⁶⁾	EPA SSL Plant	0.17	1.5	1.25	0.78
Silver	7440-22-4	6020A	39	4.2	EPA SSL Wildlife	1.4	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	--	1	ORNL Plant	0.33	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	39	2⁽⁶⁾	ORNL Plant	0.67	2.5	0.1	0.038
Zinc	7440-66-6	6010C	2300	46	EPA SSL Wildlife	15	2.5	0.5	0.27
Petroleum Hydrocarbons									
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	500	500	RIDEM Res TPH DEC	170	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	500	500	RIDEM Res TPH DEC	170	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.
2. The PSL for test pit soil is the EPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites, Residential Soil value, May 2010 (USEPA, 2010) except for GRO and ExTPH. The PSL for GRO and ExTPH is the RIDEM Residential TPH DEC (RIDEM, 2004). One-tenth RSLs are presented for non-carcinogens to correspond to a target hazard quotient of 0.1. Refer to Appendix H, Table H-1 for identification of non-carcinogens; references, and other notes applicable for specific analytes. Table H-1 also presents the RIDEM DEC, Residential Soil values (RIDEM 2004) for informational purposes.
3. The PSL for confirmatory soil is the lower of the EPA RSL, Residential Soil value (EPA RSL Res); or the selected ecological soil screening level (SSL). Appendix H, Table H-1 presents the EPA RSLs and all selected ecological SSLs and source references; and Appendix H, Table H-2 presents the ecological SSL source criteria and references. Refer also to Tables H-1 and H-2 for notes applicable for specific analytes
4. Although the PSLs are different for test pit soil and confirmatory soil, the LOQ Goals and the selected methods are the same for both types of samples, based on the lower of the two PSLs, in order to simplify sampling and analysis procedures.

Project-Specific Sampling and Analysis Plan

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5. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
6. Due to dilution factors required for analysis of solids by Method 6020A, a lower LOQ cannot be achieved by using Method 6020A; therefore, Method 6010C will be used.

Abbreviations:

- = Not available or not applicable
DEC = Direct Exposure Criteria
DL = Detection Limit
Eco = Ecological
EPA = Environmental Protection Agency
ESL = Ecological Screening Level
Ex = Extractable
GRO = Gasoline Range Organics
Invert = Invertebrate

LOD = Limit of Detection
LOQ = Limit of Quantitation
ORNL = Oak Ridge National Laboratory
Res = Residential
RIDEM = Rhode Island Department of Environmental Management
RSL = Regional Screening Level
PQL = Project Quantitation Limit
TPH = Total Petroleum Hydrocarbons

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SAP Worksheet #15b – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	8260C	0.0302	Region 3 BTAG	0.01	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	1.36	Region 3 BTAG	0.45	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.24	Region 3 BTAG	0.41	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	0.027	SCV	0.009	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	0.031	Region 3 BTAG	0.01	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	0.0165	Region 3 BTAG	0.0055	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.25	SCV	0.083	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	--	--	--	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	2.1	Region 3 BTAG	0.7	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	--	--	--	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	--	--	--	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	4.43	Region 3 BTAG	1.5	0.005	0.002	0.0007
1,4 Dichlorobenzene	106-46-7	8260C	0.599	Region 3 BTAG	0.2	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	0.27	SCV	0.09	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	0.022	SCV	0.0073	0.005	0.004	0.00083
4-Methyl-2-Pentanone	108-10-1	8260C	0.033	SCV	0.011	0.005	0.004	0.00073
Acetone	67-64-1	8260C	0.0087	SCV	0.0029	0.005	0.004	0.0016
Benzene	71-43-2	8260C	0.16	SCV	0.053	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	--	--	--	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	0.654	Region 3 BTAG	0.22	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	--	--	--	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	0.000851	Region 3 BTAG	0.00028	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.0642	Region 3 BTAG	0.021	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	0.00842	Region 3 BTAG	0.0028	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	--	--	--	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.022	SCV	0.0073	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	--	--	--	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	0.4	SCV	0.13	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	0.000051	SCV	0.000017	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	--	--	--	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	1.1	Region 3 BTAG	0.37	0.005	0.002	0.0005
Methylene Chloride	75-09-2	8260C	0.37	SCV	0.12	0.005	0.002	0.0013

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 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Styrene	100-42-5	8260C	0.559	Region 3 BTAG	0.19	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.468	Region 3 BTAG	0.16	0.005	0.002	0.00062
Toluene	108-88-3	8260C	0.05	SCV	0.017	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	1.05	Region 3 BTAG	0.35	0.005	0.002	0.00053
trans-1,3-Dichloropropene	10061-02-6	8260C	0.000051	SCV	0.000017	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	0.0969	Region 3 BTAG	0.032	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	--	--	--	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	--	--	--	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	0.16	SCV	0.053	0.005	0.002	0.00047
Semivolatile Organic Compounds								
2,4,5-Trichlorophenol	95-95-4	8270D	0.003	NOAA SQuiRT	0.001	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	0.213	Region 3 BTAG	0.071	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	0.117	Region 3 BTAG	0.039	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	0.029	Region 3 BTAG	0.0097	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	--	--	--	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	0.0416	Region 3 BTAG	0.014	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	--	--	--	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	--	--	--	0.33	0.067	0.052
2-Chlorophenol	95-57-8	8270D	0.0312	Region 3 BTAG	0.01	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	0.012	SCV	0.004	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	--	--	--	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	--	--	--	0.33	0.067	0.022
3,3'-Dichlorobenzidine	91-94-1	8270D	0.127	Region 3 BTAG	0.042	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	--	--	--	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	--	--	--	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	1.23	Region 3 BTAG	0.41	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	--	--	--	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	--	--	--	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	0.67	Region 3 BTAG	0.22	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	--	--	--	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	--	--	--	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	--	--	--	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	--	--	--	0.33	0.167	0.074
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	0.18	Region 3 BTAG	0.06	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	10.9	Region 3 BTAG	3.6	0.33	0.067	0.0063

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Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Carbazole	86-74-8	8270D	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	0.415	Region 3 BTAG	0.14	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	0.603	Region 3 BTAG	0.2	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	0.006	NOAA SQuiRT	0.002	0.33	0.067	0.0065
Di-n-butyl phthalate	84-74-2	8270D	6.47	Region 3 BTAG	2.2	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	0.061	NOAA SQuiRT	0.02	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	0.0013	NOAA SQuiRT	0.00043	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	--	--	--	0.33	0.067	0.032
Hexachloroethane	67-72-1	8270D	1.027	Region 3 BTAG	0.34	0.33	0.167	0.096
Isophorone	78-59-1	8270D	--	--	--	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	0.021	NOAA SQuiRT	0.007	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	--	--	--	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	2.68	Region 3 BTAG	0.89	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	0.504	Region 3 BTAG	0.17	0.67	0.167	0.082
Phenol	108-95-2	8270D	0.42	Region 3 BTAG	0.14	0.33	0.067	0.029
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	8270D SIM	0.0202	Region 3 BTAG	0.0067	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	0.0067	Region 3 BTAG	0.0022	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	0.0059	Region 3 BTAG	0.002	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	0.0572	Region 3 BTAG	0.019	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.108	Region 3 BTAG	0.036	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.15	Region 3 BTAG	0.05	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.13	NOAA SQuiRT	0.043	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	0.17	Region 3 BTAG	0.057	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	0.24	Region 3 BTAG	0.08	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	0.166	Region 3 BTAG	0.055	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.033	Region 3 BTAG	0.011	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	0.423	Region 3 BTAG	0.14	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	0.0774	Region 3 BTAG	0.026	0.0033	0.0033	0.00057
Indeno(1,2,3-c,d)pyrene	193-39-5	8270D SIM	0.017	Region 3 BTAG	0.0057	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	0.176	Region 3 BTAG	0.059	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	0.204	Region 3 BTAG	0.068	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	0.195	Region 3 BTAG	0.065	0.0033	0.0033	0.0017
Polychlorinated Biphenyls (PCBs)								
Aroclor-1016	12674-11-2	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0166	0.0038

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Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Aroclor-1232	11141-16-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0166	0.0039
Metals								
Aluminum	7429-90-5	6010C	25500	NOAA SQuiRT	8500	10	2.5	1.2
Antimony	7440-36-0	6010C	2	Region 3 BTAG	0.67	1	0.35	0.18
Arsenic	7440-38-2	6020A	9.8	Region 3 BTAG	3.3	0.5	0.20	0.065
Barium	7440-39-3	6010C	48	NOAA SQuiRT	16	10	0.7	0.38
Beryllium	7440-41-7	6010C	--	--	--	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	0.99	Region 3 BTAG	0.33	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	43.4	Region 3 BTAG	14	1	0.1	0.054
Cobalt	7440-48-4	6020A	50	Region 3 BTAG	17	0.5	0.20	0.089
Copper	7440-50-8	6010C	31.6	Region 3 BTAG	11	1.5	0.75	0.42
Iron	7439-89-6	6010C	20000	Region 3 BTAG	6700	10	5	2.2
Lead	7439-92-1	6010C	35.8	Region 3 BTAG	12	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	460	Region 3 BTAG	150	2.5	1	0.47
Mercury	7439-97-6	7471B	0.18	Region 3 BTAG	0.06	0.033	0.02	0.002
Nickel	7440-02-0	6010C	22.7	Region 3 BTAG	7.6	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	2	Region 3 BTAG	0.67	1.5	1.25	0.78
Silver	7440-22-4	6020A	1	Region 3 BTAG	0.33	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	--	--	--	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	57	NOAA SQuiRT	19	2.5	0.1	0.038
Zinc	7440-66-6	6010C	121	Region 3 BTAG	40	2.5	0.5	0.27
Petroleum Hydrocarbons								
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	--	--	--	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	--	--	--	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.

Project-Specific Sampling and Analysis Plan

Site Name: CED Area/QDC Outfall 001, NCBC Davisville
Project Name: Confirmatory Sampling and Drain Line Investigation
Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
Document No.: W5209575DF
Revision Number: 0
Revision Date: August 2010

2. The PSL is the selected ecological sediment screening level. The PSL references are presented in Appendix H, Table H-1, and the source criteria are presented in Appendix H-3.
3. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.

Acronyms and Abbreviations:

-- = Not available or not applicable
BTAG = Biological Technical Assistance Group
NOAA = National Oceanic and Atmospheric Administration
SCV = Secondary Chronic Value
Sed = Sediment
SQB = Sediment Quality Benchmarks
SQUIRT = Screening Quick Reference Tables
USEPA = United States Environmental Protection Agency

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SAP Worksheet #15 – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	8260C	0.0302	Sed PSL	0.01	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	0.127	CS PSL	0.042	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.1	CS PSL	0.37	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	0.027	Sed PSL	0.009	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	0.031	Sed PSL	0.01	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	0.0165	Sed PSL	0.0055	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.25	Sed PSL	0.083	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	0.89	CS PSL	0.3	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	2.1	Sed PSL	0.7	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	6.2	CS PSL	2.1	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	78	CS PSL	26	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	4.43	Sed PSL	1.5	0.005	0.002	0.0007
1,4-Dichlorobenzene	106-46-7	8260C	0.546	CS PSL	0.18	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	0.27	Sed PSL	0.09	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	0.022	Sed PSL	0.0073	0.005	0.004	0.00083
4-Methyl-2-Pentanone	108-10-1	8260C	0.033	Sed PSL	0.011	0.005	0.004	0.00073
Acetone	67-64-1	8260C	0.0087	Sed PSL	0.0029	0.005	0.004	0.0016
Benzene	71-43-2	8260C	0.16	Sed PSL	0.053	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	0.27	CS PSL	0.09	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	0.654	Sed PSL	0.22	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	0.235	CS PSL	0.078	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	0.000851	Sed PSL	0.00028	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.0642	Sed PSL	0.021	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	0.00842	Sed PSL	0.0028	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	1500	CS PSL	500	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.022	Sed PSL	0.0073	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	10.4	CS PSL	3.5	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	0.4	Sed PSL	0.13	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	0.000051	Sed PSL	0.000017	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	0.68	CS PSL	0.23	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	1.1	Sed PSL	0.37	0.005	0.002	0.0005

Project-Specific Sampling and Analysis Plan

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 Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Methylene Chloride	75-09-2	8260C	0.37	Sed PSL	0.12	0.005	0.002	0.0013
Styrene	100-42-5	8260C	0.559	Sed PSL	0.19	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.468	Sed PSL	0.16	0.005	0.002	0.00062
Toluene	108-88-3	8260C	0.05	Sed PSL	0.017	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	0.784	CS PSL	0.26	0.005	0.002	0.00053
trans-1,3-Dichloropropene	10061-02-6	8260C	0.000051	Sed PSL	0.000017	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	0.0969	Sed PSL	0.032	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	16.4	CS PSL	5.5	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	0.06	CS PSL	0.02	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	0.16	Sed PSL	0.053	0.005	0.002	0.00047
Semivolatile Organic Compounds								
2,4,5-Trichlorophenol	95-95-4	8270D	0.003	Sed PSL	0.001	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	0.213	Sed PSL	0.071	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	0.117	Sed PSL	0.039	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	0.01	CS PSL	0.0033	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	0.0609	CS PSL	0.02	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	0.0416	Sed PSL	0.014	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	0.0328	CS PSL	0.011	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	0.0122	CS PSL	0.0041	0.33	0.067	0.052
2-Chlorophenol	95-57-8	8270D	0.0312	Sed PSL	0.01	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	0.012	Sed PSL	0.004	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	61	CS PSL	20	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	1.6	CS PSL	0.53	0.33	0.067	0.022
3,3'-Dichlorobenzidine	91-94-1	8270D	0.127	Sed PSL	0.042	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	3.16	CS PSL	1.1	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	0.144	CS PSL	0.048	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	1.23	Sed PSL	0.41	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	7.95	CS PSL	2.7	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	1.1	CS PSL	0.37	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	0.67	Sed PSL	0.22	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	21.9	CS PSL	7.3	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	5.12	CS PSL	1.7	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	0.302	CS PSL	0.1	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	0.21	CS PSL	0.07	0.33	0.167	0.074

Project-Specific Sampling and Analysis Plan

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 Site Location: North Kingstown, Rhode Island

Title: Sampling and Analysis Plan
 Document No.: W5209575DF
 Revision Number: 0
 Revision Date: August 2010

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitekem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	0.18	Sed PSL	0.06	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	0.239	CS PSL	0.08	0.33	0.067	0.0063
Carbazole	86-74-8	8270D	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	0.415	Sed PSL	0.14	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	0.603	Sed PSL	0.2	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	0.006	Sed PSL	0.002	0.33	0.067	0.0065
Di-n-butyl phthalate	84-74-2	8270D	0.15	CS PSL	0.05	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	0.061	Sed PSL	0.02	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	0.0013	Sed PSL	0.00043	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	0.755	CS PSL	0.25	0.33	0.067	0.032
Hexachloroethane	67-72-1	8270D	0.596	CS PSL	0.2	0.33	0.167	0.096
Isophorone	78-59-1	8270D	139	CS PSL	46	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	0.021	Sed PSL	0.007	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	0.069	CS PSL	0.023	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	0.545	CS PSL	0.18	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	0.504	Sed PSL	0.17	0.67	0.167	0.082
Phenol	108-95-2	8270D	0.42	Sed PSL	0.14	0.33	0.067	0.029
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	8270D SIM	0.0202	Sed PSL	0.0067	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	0.0067	Sed PSL	0.0022	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	0.0059	Sed PSL	0.002	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	0.0572	Sed PSL	0.019	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.108	Sed PSL	0.036	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.015	CS PSL	0.005	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.13	Sed PSL	0.043	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	0.17	Sed PSL	0.057	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	0.24	Sed PSL	0.08	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	0.166	Sed PSL	0.055	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.015	CS PSL	0.005	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	0.423	Sed PSL	0.14	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	0.0774	Sed PSL	0.026	0.0033	0.0033	0.00057
Indeno(1,2,3-cd)pyrene	193-39-5	8270D SIM	0.017	Sed PSL	0.0057	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	0.176	Sed PSL	0.059	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	0.204	Sed PSL	0.068	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	0.195	Sed PSL	0.065	0.0033	0.0033	0.0017

Project-Specific Sampling and Analysis Plan

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Document No.: W5209575DF

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Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Polychlorinated Biphenyls (PCBs)								
Aroclor-1016	12674-11-2	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.000332	CS PSL	0.00011	0.033	0.0166	0.0038
Aroclor-1232	11141-16-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	0.000332	CS PSL	0.00011	0.033	0.0166	0.0039
TAL Metals								
Aluminum	7429-90-5	6010C	50	CS PSL	17	10	2.5	1.2
Antimony	7440-36-0	6010C	0.27 ⁽⁴⁾	CS PSL	0.09	1	0.35	0.18
Arsenic	7440-38-2	6020A	0.39	CS PSL	0.13	0.5	0.20	0.065
Barium	7440-39-3	6010C	48	Sed PSL	16	10	0.7	0.38
Beryllium	7440-41-7	6010C	10	CS PSL	3.3	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	0.36	CS PSL	0.12	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	0.29 ⁽⁴⁾	CS PSL	0.097	1	0.1	0.054
Cobalt	7440-48-4	6020A	2.3	CS PSL	0.77	0.5	0.20	0.089
Copper	7440-50-8	6010C	28	CS PSL	9.3	1.5	0.75	0.42
Iron	7439-89-6	6010C	200	CS PSL	67	10	5	2.2
Lead	7439-92-1	6010C	11	CS PSL	3.7	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	180	CS PSL	60	2.5	1	0.47
Mercury	7439-97-6	7471B	0.1	CS PSL	0.033	0.033	0.02	0.002
Nickel	7440-02-0	6010C	22.7	Sed PSL	7.6	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	0.52 ⁽⁴⁾	CS PSL	0.17	1.5	1.25	0.78
Silver	7440-22-4	6020A	1	Sed PSL	0.33	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	1	CS PSL	0.33	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	2 ⁽⁴⁾	CS PSL	0.67	2.5	0.1	0.038
Zinc	7440-66-6	6010C	46	CS PSL	15	2.5	0.5	0.27
Petroleum Hydrocarbons								

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Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitekem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	500	CS PSL	170	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	500	CS PSL	170	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.
2. PSLs are not applicable for residual material (see Section 11.2) because residual material concentrations will be compared with downgradient soil and sediment concentrations rather than with screening material. However, in order to determine the analytical methods and laboratory LOQs and LODs needed to allow comparison of residual material concentrations with detected levels of soil and sediment, values equal to the lower of the PSLs for confirmatory soil and sediment are presented. Those PSLs and their references are presented in Appendix H, Table H-1.
3. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
4. Due to dilution factors required for analysis of solids by Method 6020A, a lower LOQ cannot be achieved by using Method 6020A; therefore, Method 6010C will be used.

Abbreviations:

-- = Not available or not applicable
 CS = Confirmatory Soil
 Sed = Sediment

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

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Drain line reconnaissance/ selection of test pitting locations	Tetra Tech	Winter 2010/11	Winter 2010/11	NA	NA
Confirmatory soil sampling, sediment sampling, test pitting and sampling, and residual material sampling	Tetra Tech	Spring 2011	Spring 2011	Project Report	Spring 2011

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

Samples will be collected from the drainage pipe and downstream from the outfall in order to evaluate the nature and preliminary extent of contaminants present in environmental media as a result of Navy operations at Building 224. The sampling design was developed to address the problem statements described in Worksheet #11.

17.1 SAMPLING APPROACH

Sample collection will be focused in three general areas: a) within the 2008 soil excavation area, b) within and along the length of the drainage pipe, and c) within the downgradient wetland area. A biased sampling approach is used to target suspected sources of contamination and affected areas in order to determine whether contamination is present and to identify sources.

17.1.1 Soil Excavation Area

Five confirmatory soil samples will be collected from within the excavation area to address Problem Statement 1. Two samples will be collected from the floor of the excavated area (at a depth interval of 6 to 12 inches bgs) to determine whether contamination exceeding screening criteria extends below the excavated area, and one sample from each of the three sidewalls opposite and extending from the headwall will be collected to determine whether contamination extends horizontally beyond the excavated area. Each sample will be a composite of five soil aliquots to represent the soil in the sidewall or section of the floor. The sample locations are presented on Figure 17-1. The locations of the samples may be revised in the field based on visual or olfactory evidence of contamination.

17.1.2 Drainage Pipe

The pipe reconnaissance will be utilized to select test pitting locations. Potentially compromised sections of the pipe will be identified and targeted for test pit excavation and soil sample collection. Test pits will be excavated at up to four locations where pipe damage is observed. If fewer than four such areas are identified, locations will be selected at evenly spaced intervals approximately 400 feet apart along the drain line ending with a location at 1,200 feet, for a total of four locations. The test pit locations depicted on Figure 17-2 are spaced evenly along the length of the pipe, however these test pits will be relocated if evidence of compromised pipe is encountered during the reconnaissance.

One soil sample will be collected from each test pit for laboratory analysis. If possible, one sample of residual material present within the pipe will also be collected from compromised portions of the pipe during test pitting activities.

In addition to soil and residual material samples collected during test pitting, one sample of residual material will be collected from inside the pipe within two feet of the outfall outlet (Figure 17-1).

Analytical results from the samples of soil and residual material collected from along the length of the drainage pipe will be utilized to evaluate Problem Statements 2 and 3.

17.1.3 Downgradient Wetland Area

Five sediment samples will be collected from the wetland area to address Problem Statement 1. Samples will be collected at a depth interval of 0 to 6 inches bgs from the locations depicted on Figure 17-1. Sample locations were selected to represent the portion of the wetland that is most likely to contain the highest contaminant levels, assuming contaminants were transported to the wetland through the drainage pipe, and to evaluate the spatial distribution of contamination in sediments located within the wetland.

17.2 SAMPLING FREQUENCY

Samples will be collected during a single sampling event. Additional sampling may be necessary based on the results of the sampling proposed in this SAP. If so, a supplemental work plan will be prepared to support additional sampling that will be conducted during another field mobilization.

17.3 ANALYTICAL GROUPS

All samples will be analyzed for VOCs, GRO (C₅-C₁₂), ExTPH (C₉-C₄₀), SVOCs, PAHs, PCBs, and TAL metals. These analytes were selected either because they were detected in the soil stockpile sample collected from the outfall during waste characterization activities or were identified as contaminants-of-concern at one or more of the CED Area sites.

Samples will not be analyzed for the presence of pesticides. Pesticides were not identified as a contaminant-of-concern in the risk assessment for Sites 02 and 03. Only three pesticides (gamma-chlordane; 4,4'-DDE; and 4,4'-DDT) were detected in soil samples collected during the Remedial Investigation (RI). None of these were detected at levels approaching risk-based screening values. This

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evaluation was based on 8 surface soil samples and 15 "total soil" samples collected from Site 02 and 03 during the RI.

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SAP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates ⁽¹⁾)	Sampling SOP Reference ⁽²⁾
QF-SO-CS01 ⁽³⁾	Soil	NA	VOCs, SVOCs ⁽⁶⁾ , GRO, ExTPH, PCBs, TAL metals	1	SA-1.3
QF-SO-CS02 ⁽³⁾	Soil	NA		1	
QF-SO-CS03 ⁽³⁾	Soil	NA		1	
QF-SO-CS04 ⁽³⁾	Soil	6-12 inches bgs		1	
QF-SO-CS05 ⁽³⁾	Soil	6-12 inches bgs		1 + FD	
QF-SD01-0006 ⁽⁴⁾	Sediment	0-6 inches bgs		1	SA-1.2
QF-SD02-0006 ⁽⁴⁾	Sediment	0-6 inches bgs		1	
QF-SD03-0006 ⁽⁴⁾	Sediment	0-6 inches bgs		1	
QF-SD04-0006 ⁽⁴⁾	Sediment	0-6 inches bgs		1	
QF-SD05-0006 ⁽⁴⁾	Sediment	0-6 inches bgs		1 + FD	
QF-SO-TP01-0305 ⁽⁵⁾	Soil	3-5 feet bgs		1	SA-1.3
QF-SO-TP02-0305 ⁽⁵⁾	Soil	3-5 feet bgs		1	
QF-SO-TP03-0305 ⁽⁵⁾	Soil	3-5 feet bgs		1	
QF-SO-TP04-0305 ⁽⁵⁾	Soil	3-5 feet bgs		1 + FD	
QF-RS01	Residual Material	NA		1	See procedures in Worksheet #14 and Appendix F
QF-RS02	Residual Material	NA		1	
QF-RS03	Residual Material	NA		1	
QF-RS04	Residual Material	NA		1	
QF-RS05	Residual Material	NA		1 + FD	

- Field duplicates will be selected based on field conditions at the time of the sampling event. Field duplicates are identified here only as an example of selection at the rate of 1 per 10 samples per matrix.
- Refer to Worksheet #21 for complete reference. SOPs are included in Appendix D.
- Locations CS01 – CS03 are sidewall confirmatory samples. Locations CS04 and CS05 are excavation bottom confirmatory samples. Five-point composite samples will be collected from locations identified on Figure 17-1.. Depth of each sidewall will be measured during sample collection. Assumed 3.5 feet bgs based on field observation.
- The “0006” depth suffix represents an assumed depth interval of 0 to 6 inches below the sediment surface. The actual depth interval will be determined in the field.
- Locations TP01 – TP04 are test pit samples. The “0305” depth suffix represents an assumed depth interval of 3 to 5 feet bgs. The actual depth will be determined in the field. The depth/location of the pipe will be identified by test pitting to ensure soil samples are collected from below the pipe elevation.
- PAHs are listed as a separate group from SVOCs because PAHs will be analyzed in SIM mode (see Worksheet #15).
- Five residual material samples assumed. Residual material sample number contingent upon whether material is encountered during test pitting.

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SAP Worksheet #19 – Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference ⁽¹⁾	Containers (number, size, and type)	Sample volume ⁽²⁾ (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time ⁽³⁾ (preparation / analysis)
Soil, Sediment, and Residual Material	VOCs	5035, 8260C/Lab SOP 14	2 x 40-mL VOC Vials, preweighed, stir bar ⁽⁴⁾	5 g ⁽⁵⁾	5 mL reagent water, cool to ≤ 6°C, freeze within 48 hours	14 days to analysis
			1 x 40-mL VOC Vial, preweighed	5 g	5 mL methanol, cool to ≤ 6°C	
	GRO (C ₅ -C ₁₂)	5035, 8015D/ Lab SOP 3	1 x 40-ml VOA vial, preweighed	5 g	5 ml methanol, cool to ≤ 6°C	14 days to analysis
	ExTPH (C ₉ -C ₄₀)	3540C, 3550B, or 3570/8015D/ Lab SOPs 4, 9, 10, 11, 12	8 oz wide-mouth jar	30 g	Cool to ≤ 6°C	14 days to extraction; 40 days to analysis
	SVOCs	3540C, 3550B, or 3570/8270D/ Lab SOPs 9, 10, 11, 12, 15			Cool to ≤ 6°C	14 days to extraction; 40 days to analysis
	PAHs	3540C, 3550B, or 3570; 8270D SIM/ Lab SOPs 1, 9, 10, 12			Cool to ≤ 6°C	14 days to extraction; 40 days to analysis
	PCBs	3540C, 3550B, or 3570/8082A/ Lab SOPs 2, 9, 10, 11, 12			Cool to ≤ 6°C	Not applicable
TAL Metals	3050B, 6010C, 6020A, 7471B/ Lab SOPs 5, 6, 7, 13	4 oz wide-mouth jar	1-3 g	Cool to ≤ 6°C	180 days to analysis (ICP metals); 28 days to analysis (mercury)	

- All methods are EPA SW-846. Refer to the Analytical SOP References table (Worksheet #23) for Laboratory SOPs.
- Minimum sample volume or mass requirement.
- Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted. Per SW-846 Chapter 2, Revision 4, Feb 2007, no holding time is applicable for PCBs.
- Also include one 2-oz jar for percent moisture, cool to ≤ 6°C.
- If soil, sediment, or residual material samples are wet, extra sample (5-8 g, depending on moisture content) should be collected to achieve lower detection limits.

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SAP Worksheet #20 – Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates ¹	No. of Assigned Laboratory QC Samples ²	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks ³	No. of PT Samples	Total No. of Samples to Lab ⁴
Confirmatory Soil Sampling:									
	VOCs	5	1	1	0	0	1	0	7
	SVOC	5	1	1	0	0	0	0	6
	GRO	5	1	1	0	0	1	0	7
	ExTPH	5	1	1	0	0	0	0	6
	PAHs	5	1	1	0	0	0	0	6
	PCBs	5	1	1	0	0	0	0	6
	TAL metals	5	1	1	0	0	0	0	6
Sediment Sampling:									
	VOC	5	1	1	0	0	1	0	7
	SVOC	5	1	1	0	0	0	0	6
	GRO	5	1	1	0	0	1	0	7
	ExTPH	5	1	1	0	0	0	0	6
	PAHs	5	1	1	0	0	0	0	6
	PCBs	5	1	1	0	0	0	0	6
	TAL metals	5	1	1	0	0	0	0	6

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Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates ¹	No. of Assigned Laboratory QC Samples ²	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks ³	No. of PT Samples	Total No. of Samples to Lab ⁴
Test Pit Soil Sampling:									
	VOC	4	1	1	0	0	1	0	6
	SVOC	4	1	1	0	0	0	0	5
	GRO	4	1	1	0	0	1	0	6
	EXTPH	4	1	1	0	0	0	0	5
	PAHs	4	1	1	0	0	0	0	5
	PCBs	4	1	1	0	0	0	0	5
	TAL metals	4	1	1	0	0	0	0	5
Residual Material Sampling:									
	VOC	5	1	1	0	0	1	0	7
	SVOC	5	1	1	0	0	0	0	6
	GRO	5	1	1	0	0	1	0	7
	EXTPH	5	1	1	0	0	0	0	6
	PAHs	5	1	1	0	0	0	0	6
	PCBs	5	1	1	0	0	0	0	6
	TAL metals	5	1	1	0	0	0	0	6

Notes:

1. Collect one field duplicate per 10 field samples.
2. Assign one laboratory QC sample per 20 samples for MS/MSD analysis for organics and MS/laboratory duplicate analysis for metals.
3. Ship one trip blank per cooler with VOC and GRO samples.
4. Total number of samples does not include the laboratory QC samples.

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Following is a description of the QC sample types:

- Field QC Samples

Field duplicates will be collected by alternately filling sample containers for a given analytical group from the source being sampled. Field duplicate samples will be shipped blind to the laboratories.

Trip blanks are pre-preserved VOA vials (as described in Worksheet #19) prepared by the laboratory and shipped with the volatile (VOC and GRO) samples.

- Laboratory QC Samples - Field samples to be used for **laboratory matrix spike and matrix spike duplicate (MS/MSD)** (organic) and **matrix spike and laboratory duplicate (metals)** analyses will be assigned by the field sampler on the chain-of-custody form and sample log sheet.

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SAP Worksheet #21 – Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-05	CT-05 - Database Records and Quality Assurance; Revision 2, 2001	Tetra Tech	Not applicable	N	SOP is included in Appendix D.
HS-1.0	HS-1.0 – Utility Locating and Excavation Clearance; Revision	Tetra Tech	Remote subsurface sensing, magnetometer, ground-penetrating radar (GPR),	N	SOP is included in Appendix D.
SA-1.2	SA-1.2 – Surface Water and Sediment Sampling, Revision 5, 2008	Tetra Tech	Sampling supplies	N	SOP is included in Appendix D.
SA-1.3	SA-1.3 – Soil Sampling, Revision 9, 2008	Tetra Tech	Sampling supplies, excavator, backhoe, photoionization detector	N	SOP is included in Appendix D.
GH-1.5	GH-1.5 – Borehole and Sample Logging; Revision 1, June 1999	Tetra Tech	Not applicable	N	SOP is included in Appendix D.
SA-6.1	SA-6.1 - Non-Radiological Sample Handling ; Revision 3, 2004	Tetra Tech	Sample bottleware, packaging material, shipping materials	N	SOP is included in Appendix D.
SA-6.3	SA-6.3 - Field Documentation; Revision 3, 2009	Tetra Tech	Field logbook, field sample forms, boring logs	N	SOP is included in Appendix D.
SA-7.1	SA-7.1 - Decontamination of Field Equipment; Revision 6, 2009	Tetra Tech	Decontamination equipment (scrub brushes, phosphate-free detergent, deionized water)	N	SOP is included in Appendix D.

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SAP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity ¹	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Photo-Ionization Detector (PID)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	FOL	Operation according to manufacturer's instructions	

¹ Rental equipment and instruments will be used in the field. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs.

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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)
1	70.0033, Semivolatiles by Method 8270D SIM, Rev 5, 12/07	Definitive	Solid /PAHs	GC/MS	Mitkem Laboratories	N
2	60.0003, PCBs by Method 8082A, Rev. 8, 3/08	Definitive	Solid /PCBs	GC/ECD	Mitkem Laboratories	N
3	90.0038, Gasoline Range Organics by GC/FID using Method SW-846 8015D Rev 11, 6/10	Definitive	Solid/GRO	GC/FID	Mitkem Laboratories	N
4	60.0050, TPH by GC/FID using Method SW-846 8015D, Rev 11, 3/10	Definitive	Solid /ExTPH	GC/FID	Mitkem Laboratories	N
5	100.0111, Metals by ICP/AES Method 6010C, Rev 12, 2/09	Definitive	Solid/ICP Metals	ICP-AES	Mitkem Laboratories	N
6	100.0110, Metals in Water and Soils by ICP/MS Method 6020A, Rev 2, 4/10	Definitive	Solid/ICP Metals	ICP-MS	Mitkem Laboratories	N
7	100.0012, Mercury by Method 7470A/7471B, Rev 10, 6/10	Definitive	Solid /Mercury	CVAA	Mitkem Laboratories	N
8	110.0038, Percent Moisture, Rev 7, 2/09	Definitive	Solid/Percent Moisture	Oven	Mitkem Laboratories	N
9	50.0052, Organic Preparation of Soil Samples by Sonication, Method 3550B,, Rev 3, 2/10	Definitive	Solid/SVOCs, PAHs, PCBs, ExTPH	NA	Mitkem Laboratories	N
10	50.0053, Organic Preparation of Soil Samples by Soxhlet, Method 3540C, Rev 3, 2/10	Definitive	Solid/ SVOCs, PAHs, PCBs, ExTPH	NA	Mitkem Laboratories	N
11	50.0100, Organic Preparation of Soil Samples by MSE, Method 3570, Rev 2, 2/10	Definitive	Solid/ SVOCs, PAHs, PCBs, ExTPH	NA	Mitkem Laboratories	N
12	50.0054, Organic Extract Filtration and Concentration Techniques, Rev 2, 2/10	Definitive	Solid/ SVOCs, PAHs, PCBs, ExTPH	NA	Mitkem Laboratories	N

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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)
13	100.0104, Sample Preparation of Soils by Acid Digestion for ICP, Method 3050B, Rev 8, 3/10	Definitive	Solid/ICP Metals	NA	Mitkem Laboratories	N
14	90.0012, Volatiles by Method 8260C, Rev 11, 3/10	Definitive	Solid /VOCs	GC/MS	Mitkem Laboratories	N
15	70.0011, Semivolatiles by Method 8270D, Rev 10, 5/09	Definitive	Solid /SVOCs	GC/MS	Mitkem Laboratories	N

NA – Not applicable

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SAP Worksheet #24 – Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Note: References in this worksheet to “project-critical analytes” refer to analytes with PSLs, as identified in Worksheet #15.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
GCMS-VOCs	Initial Calibration (ICAL) - Five-point initial calibration for all analytes.	Instrument receipt, major instrument change, when CCV does not meet criteria.	<p>The average Response Factors (RFs) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorobenzene and ≥ 0.10 for chloromethane, 1,1-Dichloroethane and bromoform.</p> <p>The Percent Relative Standard Deviations (%RSDs) for RFs of Calibration Check Compound (CCCs) (as identified in SW-846 6010B) must be $\leq 30\%$, and one option below must be met for non-CCCs:</p> <p>Option 1) Per project-specific requirements, %RSDs for all non-CCCs must be $\leq 15\%$. However, up to 10% of non-CCC/SPCC, non-project-critical analytes may have $> 15\%$ RSD but $< 50\%$ RSD. If not met: Option 2) Linear least squares regression: correlation coefficient (r) must be ≥ 0.995, or Option 3) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.99 (6 points for second order).</p>	Correct problem then repeat calibration.	Analyst, Department Manager	14
	Second Source Calibration Verification (ICV)	Once after each ICAL.	Percent Recovery (%R) must be within 80-120% for all project compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL unless problem can be verified as due to ICV solution and not ICAL.	Analyst, Department Manager	

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
	Establish Retention Time (RT) Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Not applicable.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target compound must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	Continuing Calibration (CCV)	Daily before sample analysis and every 12 hours	Percent Drift or Difference (%D) must be $\leq 20\%$ for all project compounds, with allowance for a maximum of 20% of non-project critical analytes to have %D > 20% but < 50%. RFs for SPCCs must be ≥ 0.10 & ≥ 0.30 (compounds as listed above in ICAL block).	DoD project level approval must be obtained for each of the failed project-critical analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	
	BFB Tune	Prior to ICAL and at the beginning of each 12-hour clock.	Criteria listed in Section 8.1.1.2 of current revision of SOP 90.0012.	Retune and/or clean source.	Analyst, Department Manager	

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
GC/MS – Full Scan SVOCs	ICAL - A minimum 5-point calibration is required.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	The average RF for SPCCs (as identified in SW-846 6010B) must be ≥ 0.050 ; The %RSD for RFs for CCCs (as identified in SW-846 6010B) must be $\leq 30\%$, and one option below must be met for non-CCCs: Option 1) Per project-specific requirements, %RSD must be $\leq 15\%$ for all non-CCCs. However, up to 10% of non-CCC/SPCC, non-project-critical analytes may have $> 15\%$ RSD but $< 50\%$ RSD. If not met: Option 2) Linear least squares regression: r must be ≥ 0.995 , or Option 3) Non-linear regression: r^2 must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	15
	ICV (Second Source)	Once after each ICAL.	The %R must be within 80-120% for all target compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL unless problem can be verified as due to ICV solution and not ICAL.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenylphosphine (DFTPP) tune.	The RF for SPCCs must be ≥ 0.050 ; Percent Drift or Difference (%D) must be $\leq 20\%$ for all project compounds, with allowance for a maximum of 20% of non-project critical analytes to have %D $> 20\%$ but $< 50\%$.	DoD project level approval must be obtained for each of the failed project-critical analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	

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	DFTPP Tune	Prior to ICAL and every 12 hours.	Criteria listed in Section 8.2.2, of current revision of SOPs 70.0011 and 70.0033.	Retune and/or clean source.	Analyst, Department Manager	
GC/MS –SIM PAHs	ICAL - A minimum 5-point calibration is required.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	Project-specific criteria: The average RF for all target compounds must be ≥ 0.050 . The %RSD for all target compounds must be $\leq 20\%$. If not met, Option 1 or Option 2 below must be met: Option 1) Linear least squares regression: $r \geq 0.995$ Option 2) Non-linear regression: $r^2 \geq 0.99$ (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	1
	ICV (Second Source)	Once after each ICAL.	The %R must be within 80-120% for all target compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL unless problem can be verified as due to ICV solution and not ICAL.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenylphosphine (DFTPP) tune.	Project-specific criteria: The RF for all target compounds must be ≥ 0.050 . The %D for all target compounds and surrogates must be $\leq 20\%D$. (D = Difference or Drift)	DoD project level approval must be obtained for each of the failed project-critical analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	

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	DFTPP Tune	Prior to ICAL and every 12 hours.	Criteria listed in Section 8.2.2, of current revision of SOPs 70.0011 and 70.0033.	Retune and/or clean source.	Analyst, Department Manager	
GC/FID GRO, ExTPH	Initial Calibration	After major instrument maintenance, or when CCV fails	5 point calibration curve for individual compounds, with average %Relative Standard Deviation for the hydrocarbon range $\leq 20\%$	Check instrument performance, maintenance, recalibrate	Analyst, Department Manager	3, 4
	ICV (Second Source)	Once after each ICAL. For GRO, the laboratory control sample (LCS) in each batch serves as the ICV, as it is a second-source, non-prepared standard.	The %R must be within 80-120% for the hydrocarbon range.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL unless problem can be verified as due to ICV solution and not ICAL.	Analyst, Department Manager	
	Continuing Calibration	Every 12 hours and/or every 20 samples, and at end of sequence.	%Difference for the hydrocarbon range must be $\leq 20\%$ (GRO) or $\leq 25\%$ (ExTPH).	Check instrument performance, maintenance, recalibrate	Analyst, Department Manager	
ICP-AES	ICAL - 3 point calibration plus blank	Daily prior to sample analysis.	Correlation coefficient (r) must be ≥ 0.995 .	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	5
	ICV (Second Source)	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% of true values.	Do not use results for failing elements unless the ICV $> 110\%$ and the sample results are non-detect. Investigate and correct problem.	Analyst, Department Manager	

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
	Calibration Blanks (initial and continuing)	Before beginning a sample sequence, after every 10 field samples and at end of the analysis sequence.	Absolute value must be \leq LOD for all target analytes.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	After every 10 field samples and at the end of each run sequence.	%R must be within 90-110% of true values.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
ICP-MS	Tune	Daily prior to calibration.	Mass calibration must be within 0.1 atomic mass unit (amu) from the true value. Resolution must be <0.9 amu full width at 10% peak height. Five integrations %RSD must be $<5\%$.	Perform necessary equipment maintenance.	Analyst, Department Manager	6
	ICAL - 3 point calibration plus blank	Daily prior to sample analysis.	Correlation coefficient (r) must be ≥ 0.995 .	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	
	ICV (Second Source)	Once after each ICAL, and before beginning a sample run.	%R must be within 90-110% of true values.	Do not use results for failing elements unless the ICV $> 110\%$ and the sample results are non-detect. Investigate and correct problem.	Analyst, Department Manager	
	Calibration Blanks (initial and continuing)	Before beginning a sample sequence, after every 10 field samples and at end of the analysis sequence.	Absolute value must be \leq LOD for all target analytes.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	

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	CCV	After every 10 field samples and at the end of each run sequence.	%R must be within 90-110% of true values.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
Mercury analyzer	ICAL - 5 point calibration plus a blank	Upon instrument receipt, major instrument change, at the start of each day.	Correlation coefficient (r) must be ≥ 0.995 .	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	7
	ICV (Second Source)	Once after each ICAL, prior to beginning a sample run.	%R must be within 90-110% of the true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample sequence, after every 10 field samples and at end of the analysis sequence. For negative blanks, absolute value < LOD.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	
	CCV	Beginning and end of each run sequence and every 10 field samples.	%R must be within 80-120% of the true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
GC/ECD-PCBs	ICAL - five-point calibration.	Instrument receipt, major instrument change, when CCV does not meet criteria.	Five point calibration of Aroclors 1016/1260, 1242, 1248, and 1254 – One of the options below: Option 1: %RSD for each analyte must be ≤ 20%; Option 2: linear least squares regression: r must be ≥ 0.995; Option 3: non-linear regression: r ² must be ≥ 0.99 (6 points shall be used for second order) Mid-point calibration of Aroclors 1221 and 1232; if targets are detected, then 5-point calibration is performed.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	2
	ICV	Immediately following ICAL.	%R must be within 80-120% for all project target analytes.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL unless problem can be verified as due to ICV solution and not ICAL.	Analyst, Department Manager	
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	%Difference or %Drift must ≤ 20%.	DoD project level approval must be obtained for each of the failed project-critical analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	

1. Refer to the Analytical SOP References table (Worksheet #23).

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

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ¹
GC/MS - VOCs	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.	Tune (BFB), CCV	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Tune daily, CCV at the beginning of each 12-hour shift.	Acceptable tune, Acceptable CCV.	See Section 6.2 in SOP for CA details. Repeat tune, calibration or CCV and any affected samples.	Analyst, Department Manager	14
GCMS – Full Scan SVOCs	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner and replace septum as needed, replace Electron Multiplier	Tune (DFTPP), CCV	Monitor instrument performance via tuning mass criteria, and CCV.	Tune daily, CCV at the beginning of each 12-hour shift.	Acceptable tune, Acceptable CCV.	See Section 6.2 in SOP for CA details. Repeat tune, calibration or CCV and any affected samples.	Analyst, Department Manager	15
GCMS – SIM PAH	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner and replace septum as needed, replace Electron Multiplier	Tune (DFTPP), CCV	Monitor instrument performance via tuning mass criteria, and CCV.	Tune daily, CCV at the beginning of each 12-hour shift.	Acceptable tune, Acceptable CCV.	See Section 6.2 in SOP for CA details. Repeat tune, calibration or CCV and any affected samples.	Analyst, Department Manager	1
ICP-AES	Perform leak test, change pump tubing, change torch and window, clean filters	Calibration Verification and Calibration Blank	Monitor instrument performance via CCV/CCB.	Daily, after every 10 field samples	Acceptable CCV/CCB.	See Section 6.2 in SOP for CA details. Repeat calibration or CCV and any affected samples.	Analyst, Department Manager	5
ICP-MS	Perform leak test, change pump tubing, remove and clean cone, extraction lens and ion lens stack	Calibration Verification and Calibration Blank	Monitor instrument performance via CCV/CCB.	Daily, after every 10 field samples	Acceptable CCV/CCB.	See Section 6.2.2 in SOP for CA details. Repeat tune, calibration or CCV and any affected samples.	Analyst, Department Manager	6
Mercury Analyzer	Perform leak test, change tubing, clean window, clean filters	Initial Calibration Verification and Initial Calibration Blank	Monitor instrument performance via ICV/ICB.	Daily, after every 10 field samples	Acceptable ICV/ICB.	Replace connections, replace pump tubing, clean all filters. Repeat calibration or CCV.	Analyst, Department Manager	7

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Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference¹
GC/ECD – PCBs; GC/FID – GRO, ExTPH	Check for leaks, replace gas line filters, clip end of column, recondition or replace column, clean injection port/liner, replace septum	CCV	Monitor instrument performance via CCV.	Daily, after every 10 field samples	Acceptable CCV.	See Attachment 1 of SOP for CA details. Repeat calibration or CCV and any affected samples.	Analyst, Department Manager	2, 3, 4

1. Refer to the Analytical SOP References table (Worksheet #23).

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SAP Worksheet #26 – Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System - Mitkem Laboratories

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operations Leader, Tetra Tech
Sample Packaging (Personnel/Organization): Field Operations Leader, Tetra Tech
Coordination of Shipment (Personnel/Organization): Field Operations Leader, Tetra Tech
Type of Shipment/Carrier: Laboratory courier service (Mitkem)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff (organic / inorganic), Mitkem Laboratories
Sample Determinative Analysis (Personnel/Organization): GC/MS, Metals Laboratory Staff, Mitkem Laboratories
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from submittal of final data report
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 6 months from submittal of final data report
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians, Mitkem Laboratories

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

Sample Designation and Tracking System

Each sample collected will be assigned a unique sample tracking number used to catalog the results. The planned sample tracking numbers are listed in Worksheet #18. If more samples are added, the same labeling patterns will be followed. Any other pertinent information regarding sample identification will be recorded on the sample log sheets, chains of custody forms or in the field logbooks.

Field QC sample types are presented in Worksheet #20. Field QC samples will be designated using the following identifiers:

Site: "QF" for Site QDC Outfall 001

Medium: "SO" for soil samples (confirmatory and test pit)
"SD" for sediment samples
"RS" for the residual material samples

Field QC designations will conform to the following formats:

- Field Duplicates: Blind field duplicate sample designations will consist of the site and medium identifiers, the label "DUP", a sequential value (the nth duplicate sample collected for that medium during that sampling event) and a date (MMDDYY). The sample log sheet will note from which sample location the duplicate was collected. For example, a soil confirmatory sample field duplicate collected on July 12, 2010, would be labeled QF-SO-DUP01-071210. A test pit soil sample field duplicate collected on July 14, 2010, would be labeled QF-SO-DUP02-071410
- Trip Blanks: Trip blank designations will consist of the site, the medium of the samples associated with the trip blank, the label "TB", a sequential value (the nth trip blank collected for that medium during that sampling event) and the date (MMDDYY). For example, the first trip blank for the sampling event shipped with soil samples on July 12, 2010 will be QF-SO-TB01-071210. The trip blank to be shipped with the residual material samples on August 12, 2010 will be QF-RS-TB01-081210.

Laboratory QC samples (matrix spike and laboratory duplicate samples) have no separate sample identifier codes, but are assigned on the chain-of-custody record and sample log sheet.

Sample Handling and Chain-of Custody Procedures

Custody of samples must be maintained and documented at all times. To ensure the integrity of a sample from collection through analysis, an accurate written record is necessary to trace the possession and handling of the sample. This documentation is referred to as the "chain of custody" form. Chain of custody begins when samples are collected in the field, and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be accompanied by a chain-of-custody form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

Following collection, samples will be placed on ice in a secure cooler and attended by Tetra Tech personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. Chain of custody procedures are described in further detail in the following Tetra Tech SOPs:

- SA-6.3 Field Documentation
- SA-6.1 Non-Radiological Sample Handling

The samples will be shipped to the laboratories in coolers packed with ice and bubble wrap, or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain of custody is maintained. The chain-of-custody forms will be shipped to the laboratory with the samples.

Samples will be shipped to the laboratories by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times before sample extraction, digestion, or analysis are presented in Worksheet #19. Saturday deliveries will be coordinated by the FOL or his or her designee with the laboratory. Worksheet #19 also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

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Each sample collected will be assigned a unique sampling tracking number. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the chain-of-custody form. Preservatives used will be stated on the sample label and the chain of custody form. The chain-of-custody form is a two-part form: the original accompanies the samples to the analytical laboratory, and the copy is retained by the sampling staff until it is submitted to the project manager and data validators.

One copy of the chain-of-custody form will be kept by archive in the project files. Information to be recorded on the chain-of-custody form should include:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- Quality control (QC) sample designation
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of individual releasing the shipping container

Laboratory custody procedures are addressed in Mitkem SOPs 30.0003 and 30.0024, included in Appendix G.

The field crew will attempt to identify any potentially high concentration samples on the chain-of-custody form.

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

Note: Mitkem's statistically-derived limits referenced in Worksheet #28 are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Note: References in Worksheet #28 to "project-critical analytes" refer to analytes with PSLs, as identified in Worksheet #15.

Matrix	Soil, Sediment, Residual Material					
Analytical Group	VOCs					
Analytical Method/ SOP Reference	SW846 8260C/ SOP 90.0012					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Project-specific limits: Contaminants in the method blank must be < 1/2 LOQ, except common lab contaminants, which must be <LOQ. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Reprepare and reanalyze the method blank and all associated samples with results > LOD for project-critical analytes. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	Four per sample: Dibromofluoro methane, 1,2-dichloroethane -d4, Toluene-d8, BFB.	%Rs must meet the laboratory statistically-derived control limits. Current limits are provided in <i>Appendix G</i> .	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic or matrix interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Laboratory Department Manager and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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Matrix	Soil, Sediment, Residual Material					
Analytical Group	VOCs					
Analytical Method/ SOP Reference	SW846 8260C/ SOP 90.0012					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Laboratory Control Sample (LCS) Laboratory Control Sample Duplicate (LCSD)	One per preparation batch of 20 or fewer samples of similar matrix. One LCSD per prep batch of twenty or fewer samples of similar matrix if no MS/MSD in batch.	%Rs must meet the laboratory statistically- derived control limits. Current limits are provided <i>in Appendix G</i> . Refer to DoD QSM Version 4.1 (QSM) Table G-1 for marginal exceedance criteria. The RPD between LCS and LCSD must be \leq 30%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed project critical analytes, if sufficient sample material is available. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accura cy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	%Rs should meet the laboratory statistically- derived control limits. Current limits are provided <i>in Appendix G</i> . The RPD between MS and MSD should be \leq 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Internal Standards (IS)	Three per sample- Fluorobenzene , Chlorobenzene -d5, and 1,4- Dichlorobenze ne-d4.	Retention times for internal standards must be \pm 30 seconds and the responses within -50% to +100% of the ICAL midpoint standard.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning unless obvious chromatographic or matrix interference.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

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

Matrix	Soil, Sediment, Residual Material					
Analytical Group	SVOCs – Full Scan and SIM					
Analytical Method/ SOP Reference	SW846 8270D / SOP 70.0011 SW846 8270D SIM/ SOP 70.0033					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Project-specific limits: Contaminants in the method blank must be < ½ LOQ, except common lab contaminants, which must be <LOQ. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Reprepare and reanalyze the method blank and all associated samples with results > LOD for project-critical analytes. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	Six for <u>Full Scan</u> : 2,4,6-Tribromophenol, 2-Fluorobiphenyl, 2-Fluorophenol, Nitrobenzene-d5, Phenol-d5 and Terphenyl-d14. One for <u>SIM</u> : Benzo (e) pyrene.	%Rs must meet the laboratory statistically-derived control limits. Current limits are provided <i>in Appendix G</i> .	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic or matrix interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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Title: Sampling and Analysis Plan
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Matrix	Soil, Sediment, Residual Material					
Analytical Group	SVOCs – Full Scan and SIM					
Analytical Method/ SOP Reference	SW846 8270D / SOP 70.0011 SW846 8270D SIM/ SOP 70.0033					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One per preparation batch of 20 or fewer samples of similar matrix. One LCSD per prep batch of twenty or fewer samples of similar matrix if no MS/MSD in batch.	%Rs must be within laboratory statistically derived limits. Current limits are provided in <i>Appendix G</i> . Refer to DoD QSM Version 4.1 (QSM) Table G-1 for marginal exceedance criteria. RPD must be ≤ 30%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed project critical analytes, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	%Rs should be within laboratory statistically derived limits. Current limits are provided in <i>Appendix G</i> . RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	<u>Full Scan</u> uses six per sample –1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12. <u>SIM</u> uses five of the above (not 1,4-DCB-d4).	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of the ICAL midpoint.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning unless obvious chromatographic or matrix interference.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

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

Matrix	Soil, Sediment, Residual Material					
Analytical Group	PCBs					
Analytical Method/ SOP Reference	SW846 8082A / SOP 60.0003					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Project-specific limits: Contaminants in the method blank must be < 1/2 LOQ, except common lab contaminants, which must be <LOQ. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Reprepare and reanalyze the method blank and all associated samples with results > LOD for project-critical analytes. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample: Decachloro-biphenyl. (Tetrachloro-m-xylene [TCX] is included as an alternate surrogate.)	Project-specific limits: Decachloro-biphenyl %Rs must meet the laboratory statistically-derived control limits. Current limits are provided <i>in Appendix G</i> .	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic or matrix interference with surrogate is present, reanalysis may not be necessary. Monitor TCX recovery to assist in sample evaluation.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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Matrix	Soil, Sediment, Residual Material					
Analytical Group	PCBs					
Analytical Method/ SOP Reference	SW846 8082A / SOP 60.0003					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One per preparation batch of 20 or fewer samples of similar matrix. One LCSD per prep batch of twenty or fewer samples of similar matrix if no MS/MSD in batch.	%R of 1016/1260 must be within laboratory statistically derived limits. Current limits are provided <i>in Appendix G</i> . Refer to DoD QSM Version 4.1 (QSM) Table G-1 for marginal exceedance criteria. RPD must be ≤ 30%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed project critical analytes, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	%R of 1016/1260 should be within laboratory statistically derived limits. Current limits are provided <i>in Appendix G</i> . RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/ Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

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Matrix	Soil, Sediment, Residual Material					
Analytical Group	PCBs					
Analytical Method/ SOP Reference	SW846 8082A / SOP 60.0003					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second Column Confirmation	All positive results must be confirmed.	Project-specific requirement: RPD should be $\leq 40\%$. Project-specific reporting requirements: The laboratory should designate and always use the same chromatographic stationary phase for Column 1 and likewise for Column 2. Results should be reported from Column 1. However, if the between-column RPD exceeds 40%, the analyst must select which result (i.e., from Column 1 or Column 2) to report; and the laboratory must provide an explanation in the case narrative why the particular result was selected for each affected target analyte.	None. Apply qualifier if RPD $>40\%$ and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

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

Matrix	Soil/Sediment/Residual Material					
Analytical Group	GRO					
Analytical Method/ SOP Reference	SW846 8015D/ SOP 90.0038					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of 20 or fewer samples of similar matrix	Project-specific limits: Contaminants in the method blank must be < ½ LOQ. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Reprepare and reanalyze the method blank and all associated samples with results > LOD for project-critical analytes. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per prep batch of 20 or fewer samples of similar matrix	The hydrocarbon range %R must be within 80-120%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time. If the LCS recovery is high but the sample results are < LOQ, narrate. Flag with * on Form 3.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCSD	One per prep batch of 20 or fewer samples of similar matrix if no MS/MSD in batch	The hydrocarbon range %R must be within 80-120 %. RPD must be ≤ 20%	Same as for LCS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Precision	Same as Method/SOP QC Acceptance Limits.

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Matrix	Soil/Sediment/Residual Material					
Analytical Group	GRO					
Analytical Method/ SOP Reference	SW846 8015D/ SOP 90.0038					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	The hydrocarbon range %R should be within 60-140%.	If recovery is outside limits and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable check standard preparation. Speak with PM regarding further action. Flag outliers with * on Form 3.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike Duplicate	One per batch of 20 field samples of similar matrix as identified by sampler	The hydrocarbon range %R should be within 60-140%. RPD should be \leq 30%.	Same as for MS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Precision	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample: BFB.	%Rs must meet the laboratory statistically-derived control limits. Current limits are provided <i>in Appendix G</i> .	Unless obvious chromatographic or matrix interference, if sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag with * on Form 2.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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

Matrix	Soil/Sediment/Residual Material					
Analytical Group	ExTPH					
Analytical Method/ SOP Reference	SW846 8015D/ SOP 60.0050					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of 20 or fewer samples of similar matrix	Project-specific limits: Contaminants in the method blank must be < ½ LOQ. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Reprep and reanalyze the method blank and all associated samples with results > LOD for project-critical analytes. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per prep batch of 20 or fewer samples of similar matrix	The hydrocarbon range %R must be within 60-140%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time. If the LCS recovery is high but the sample results are < LOQ, narrate. Flag with * on Form 3	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCSD	One per prep batch of twenty or fewer samples of similar matrix if no MS/MSD in batch.	The hydrocarbon range %R must be within 60-140%. RPD must be ≤ 20%.	Same as for LCS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Precision	Same as Method/SOP QC Acceptance Limits.
MS	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	The hydrocarbon range %R should be within 50-150%.	If recovery is outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable check standard preparation. Speak with PM regarding further action. Flag outliers with * on Form 3.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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Matrix	Soil/Sediment/Residual Material					
Analytical Group	ExTPH					
Analytical Method/ SOP Reference	SW846 8015D/ SOP 60.0050					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MSD	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	The hydrocarbon range %R should be within 50-150%. RPD should be \leq 30%.	Same as for MS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias, Precision	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample: O-terphenyl	%Rs must meet the laboratory statistically-derived control limits. Current limits are provided <i>in</i> <i>Appendix G.</i>	Unless obvious chromatographic or matrix interference, if sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag with * on Form 2.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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

Matrix	Soil, Sediment, Residual Material					
Analytical Group	Metals (Including Mercury)					
Analytical Method/SOP Reference	SW-846 6010C/6020A and 7471A/ SOPs 100.0111, 100.0110, 100.0012					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	Project-specific limits: Contaminants in the method blank must be < 1/2 LOQ. For negative blanks, the absolute value must be < LOD. However, contaminant concentrations are acceptable if they are < the greater of 1/10 the amount measured in any sample or 1/10 the PSL.	Correct the problem. Report sample results that are <LOD. Re-prepare and reanalyze the method blank and all associated samples with results > LOD	Analyst, Laboratory Department Manager, and Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
IS (ICP-MS only)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	For each sample, IS intensity within 30-120% of that of initial calibration standard.	Reanalyze the sample at 5-fold dilution with addition of appropriate amounts of internal standards.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples of the same matrix.	%Rs must be within 80-120% of the true value.	Re-digest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	%RPD should be < 20%.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	If values are ≥ 5x LOQ, RPD should be ≤ 35%; if values are < 5x LOQ, Absolute Difference should be ≤ 2x LOQ.

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Matrix	Soil, Sediment, Residual Material					
Analytical Group	Metals (Including Mercury)					
Analytical Method/SOP Reference	SW-846 6010C/6020A and 7471A/ SOPs 100.0111, 100.0110, 100.0012					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS	One per sample delivery group (SDG) or every 20 samples as identified by sampler.	%R should be within 80-120% if sample concentration is < 4x spike added. Project-specific reporting requirement: Report %R for all metals.	Flag results with "N" for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	%R should be within 75-125%
ICP Interference Check Sample - ICSA & ICSB (for ICP only)	Daily, before sample injections	ICSA %Rs must be less than the absolute value of the LOD and ICSB %Rs must be within 80-120% of the true value.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst, Department Manager	Accuracy	ICSA - ICP-AES: If sample interferent concentrations are > 50% ICSA interferent concentrations, absolute value of non-interferent ICSA must be within true value ± LOD. ICP-MS: Absolute value of ICSA must be within true value ± LOQ. ICSB - %R must be within 80-120% of the true value.
Serial Dilution (SD) (for ICP only)	One SD (5x) is performed for each batch of 20 samples of the same matrix.	If the original sample result is at least 50x DL, the five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike addition.	Analyst, Laboratory Department Manager, and Data Validator	Precision	If the original sample result is at least 50x LOQ, the five-fold dilution must agree within ± 10% of the original measurement

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Analytical Group	Metals (Including Mercury)					
Analytical Method/SOP Reference	SW-846 6010C/6020A and 7471A/ SOPs 100.0111, 100.0110, 100.0012					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post-Digestion Spike (for ICP only)	Project-specific frequency: When MS recovery fails or analyte concentration in all samples < 50x LOD	%R should be within 75-125%.	Qualify results and note in narrative.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

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

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain-of-Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Drilling Logs Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Wilmington, Massachusetts office.
Laboratory Documents and Records - in the form of an analytical data package: Sample receipt/login form Sample storage records Sample preparation logs Equipment calibration logs Sample analysis run logs Reported results for standards, QC checks, and QC samples Data completeness checklists Telephone logs Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and electronic deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Wilmington, Massachusetts project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (include tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Wilmington, Massachusetts project file.
Reports Data Report	All versions of the Project Report and support documents (e.g., Data Validation Reports) will be stored in hard copy in the Tetra Tech Wilmington, Massachusetts project file and electronically in the server library.

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

Matrix	Analytical Group	Sample Locations/ ID Number	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)
Soil, Sediment, and Residual Material	VOCs	See Worksheet #18	EPA SW-846 Method 8260C	21 days	Mitkem Laboratories 175 Metro Center Boulevard Warwick, Rhode Island 02886-1755 Contact: Edward Lawler Laboratory Operations Manager 401-732-3400, ext. 315	Not applicable
	SVOCs		EPA SW-846 Method 8270D			
	PAHs		EPA SW-846 Method 8270D SIM			
	PCBs		EPA SW-846 Method 8082A			
	GRO		EPA SW-846 Method 8015D			
	ExTPH		EPA SW-846 Method 8015D			
	TAL Metals		EPA SW-846 Methods 6010C/6020A/7471B			

Data package deliverables are detailed in the Analytical Technical Specifications included in Appendix E. Data packages will be provided as both hardcopy and portable document format (.PDF). Laboratories will provide a Naval Installation Restoration Information Solutions (NIRIS) compatible electronic data deliverable (EDD). Data packages will be Contract Laboratory Program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). Data will be stored by the analytical laboratory for five years.

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

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Mitkem QA Manager	Mitkem QA Manager	DoD ELAP Accrediting Body Auditor

Note: Mitkem is DoD Environmental Laboratory Accreditation Program (DoD ELAP) accredited. The DoD ELAP certificate of accreditation and Mitkem's Rhode Island certification are included in Appendix G.

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SAP Worksheet #32 – Assessment Findings and Corrective Action 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 Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Laboratory Manager, QAM	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP

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

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per sample delivery group (SDG)	Within 3 weeks of receipt of laboratory data	Project Chemist (Lucy Guzman), Tetra Tech	PM (Scott Anderson), Tetra Tech Tetra Tech project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem (on the same day)	QAM (Tom Johnston), Tetra Tech	PM (Tetra Tech), QAM (Tetra Tech), PM (Tetra Tech), Tetra Tech project file
Project monthly progress report	Monthly for duration of the project	Monthly	PM (Scott Anderson), Tetra Tech	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling is occurring	FOL (Michael Alroy), Tetra Tech	PM (Tetra Tech)
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (same day)	Subcontracted laboratory QAM	Tetra Tech project file

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 PM, and the Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	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, Mitkem2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	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 actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	Internal	Tetra Tech, FOL or designee
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is significantly out of control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Mitkem
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech

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Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Analytical Data Packages	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	Laboratory QAM, Mitkem
Electronic Data Deliverables (EDDs)/ Analytical Data Packages	Each EDD will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech
Electronic Data Deliverables (EDDs)/ Analytical Data Packages	Each data package will be verified for completeness by the Tetra Tech Data Validator. Missing information will be requested by the Tetra Tech Data Validator from the Laboratory PM.	External	Data Validators, Tetra Tech

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. 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.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	<p>Accuracy - Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs 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.</p> <p>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/MSDs; and LCS/ LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.</p> <p>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.</p> <p>Completeness - Review the chain-of-custody forms 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. The Tetra Tech Data 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 (SQL) database.</p>	Project Chemist or Data Validators, Tetra Tech

SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table (Continued)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		PSLs - Discuss the impact on reported DLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PSLs to the SQL database. Flag samples and notify the Tetra Tech PM of samples that exceed PSLs listed in Worksheet #15.	Project Chemist or Data Validators, and Data Manager, Tetra Tech
		QA/QC - 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 QAM shall have contacted the Tetra Tech PM.	Project Chemist or Data Validators, Tetra Tech
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Project Chemist or Data Validators, Tetra Tech

SAP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table
 (UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil, Sediment, and Residual Material	VOCs, SVOCs, PAHs, GRO, ExTPH	Tier II ⁽¹⁾ data validation. Project-specific criteria for VOCs by SW-846 8260C, SVOCs by SW-846 8270D, PAHs by SW-846 8270D SIM, and GRO and ExTPH by SW-846 8015D are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (USEPA, 1996) will be applied using these criteria.	Tetra Tech, Project Chemist (L. Guzman) and staff chemists
IIa and IIb	Soil, Sediment, and Residual Material	PCBs	Tier II ⁽¹⁾ data validation. Project-specific criteria for PCBs by SW-846 8082A are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (USEPA, 2004) will be applied using these criteria.	Tetra Tech, Project Chemist (L. Guzman) and staff chemists
IIa and IIb	Soil, Sediment, and Residual Material	TAL Metals	Tier II ⁽¹⁾ data validation. Project-specific criteria for metals by SW-846 6010C/6020A/7471B are listed in Worksheets #12, #15, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, November 2008 (USEPA, 2008) will be applied using these criteria.	Tetra Tech, Project Chemist (L. Guzman) and staff chemists

1 – As defined in the Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, Attachment B, “Region 1 Tiered Organic and Inorganic Data Validation Guidelines”, July 1, 1993, Draft (USEPA, 1993).

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 these data characteristics:

Completeness: The FOL acting on behalf of the Project Team will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Tetra Tech PM determines that the deviations compromise the ability to meet project objectives she will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision: The 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 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, matrix spike, matrix spike duplicate, and laboratory control samples. 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.

Representativeness: A project scientist identified by the Tetra Tech PM 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 analyzed in accordance with this 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 Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property 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 the Project Chemist indicates that such quantitative analysis is required.

Sensitivity: The 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.

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 Project Manager will assess whether the data collectively support the attainment of project objectives. They 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 them in a weight-of-evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical evaluations, non-detected values will be represented by a concentration equal to one-half the LOD.

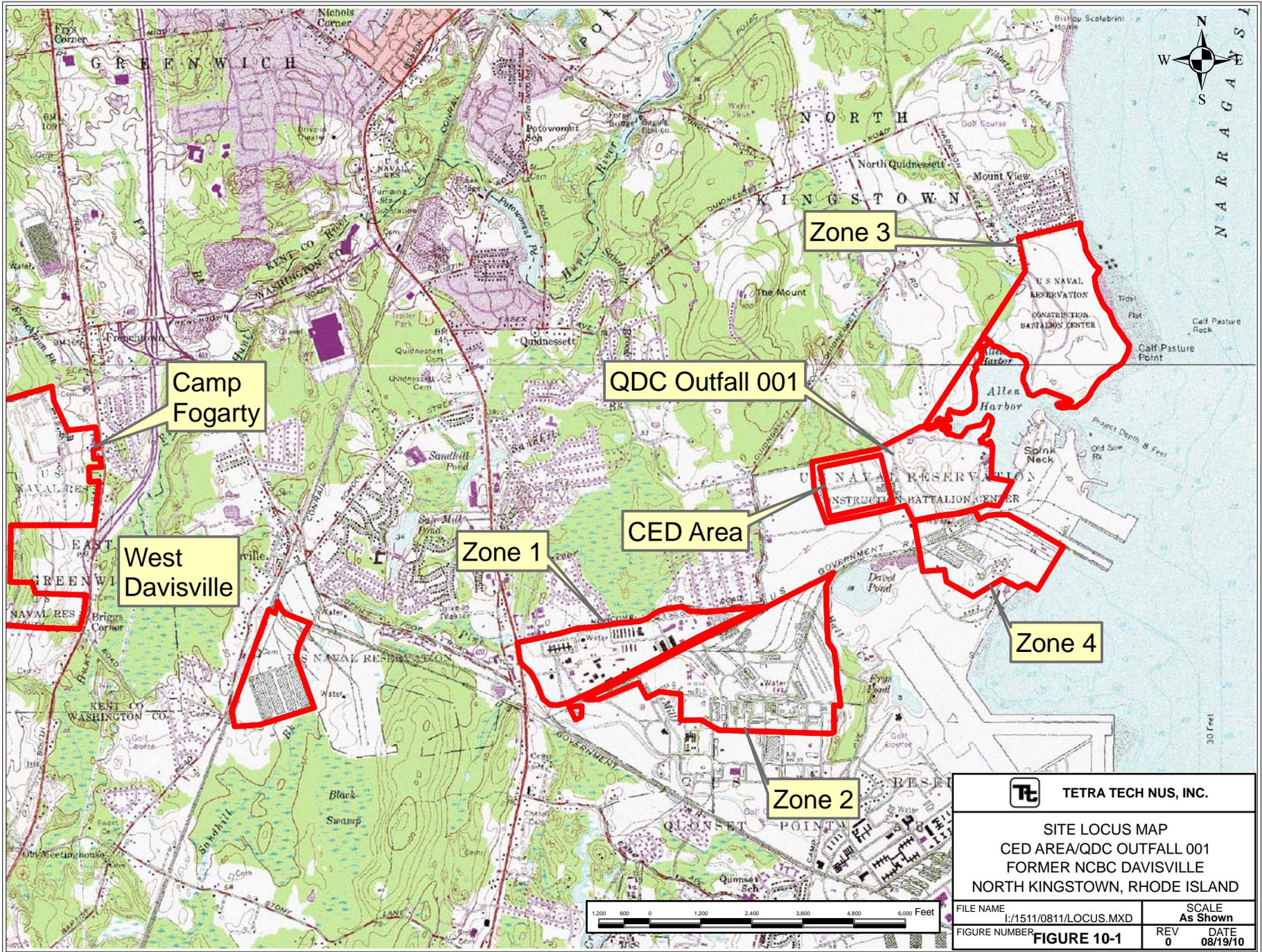
Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, and FOL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or 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). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.

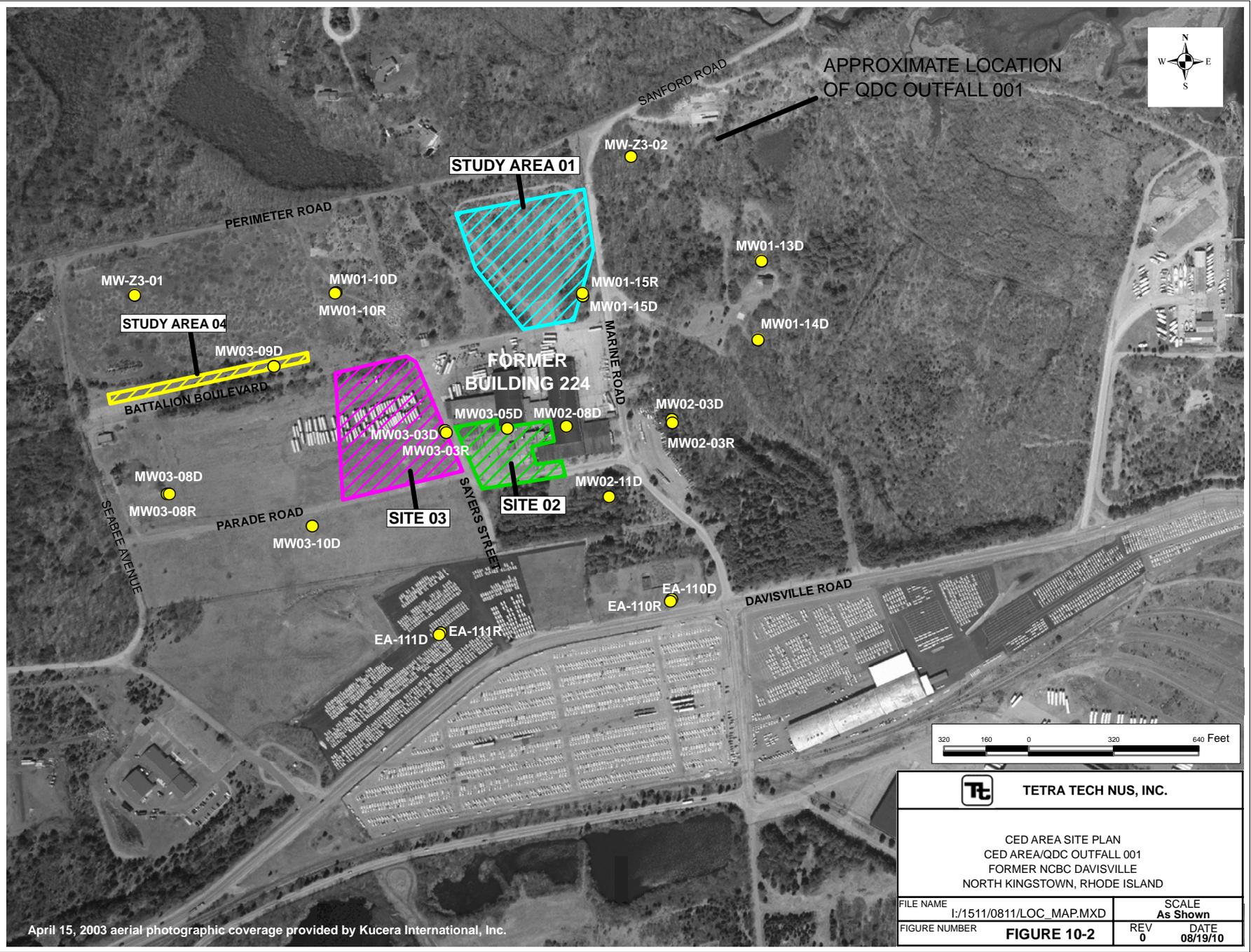
FIGURES



 TETRA TECH NUS, INC.	
SITE LOCUS MAP CED AREA/QDC OUTFALL 001 FORMER NCBC DAVISVILLE NORTH KINGSTOWN, RHODE ISLAND	
FILE NAME	SCALE
I:\1511\0811\LOCUS.MXD	As Shown
FIGURE NUMBER	REV DATE
FIGURE 10-1	0 08/19/10



APPROXIMATE LOCATION OF QDC OUTFALL 001



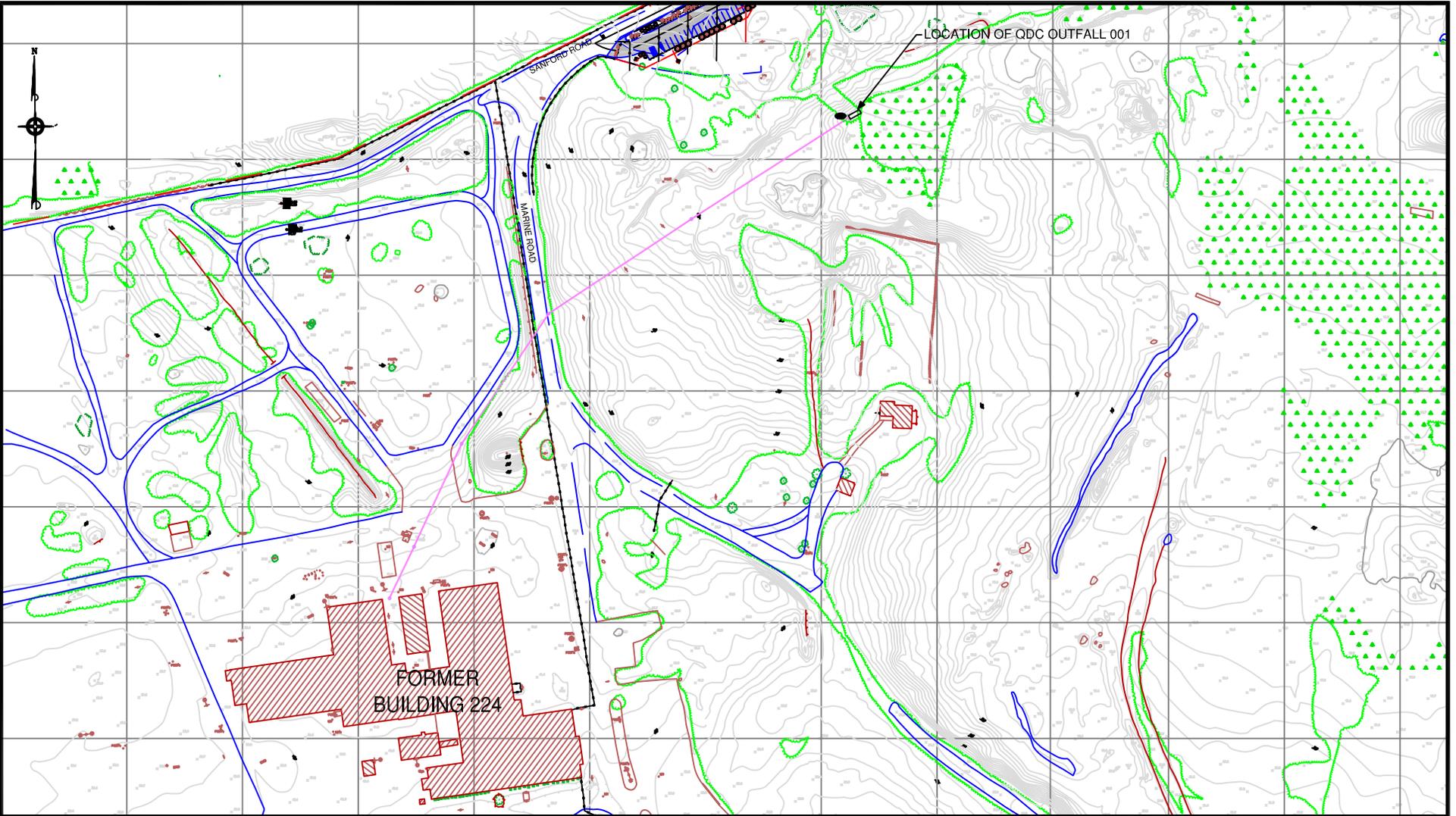
April 15, 2003 aerial photographic coverage provided by Kucera International, Inc.



Tt TETRA TECH NUS, INC.

CED AREA SITE PLAN
CED AREA/QDC OUTFALL 001
FORMER NCBC DAVISVILLE
NORTH KINGSTOWN, RHODE ISLAND

FILE NAME	I:/1511/0811/LOC_MAP.MXD	SCALE	As Shown
FIGURE NUMBER	FIGURE 10-2	REV	DATE
		0	08/19/10

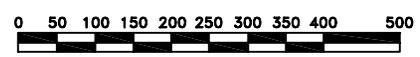


Legend:

 - Limits of soil stockpile

Scale:

Scale in feet



TETRA TECH NUS, INC.

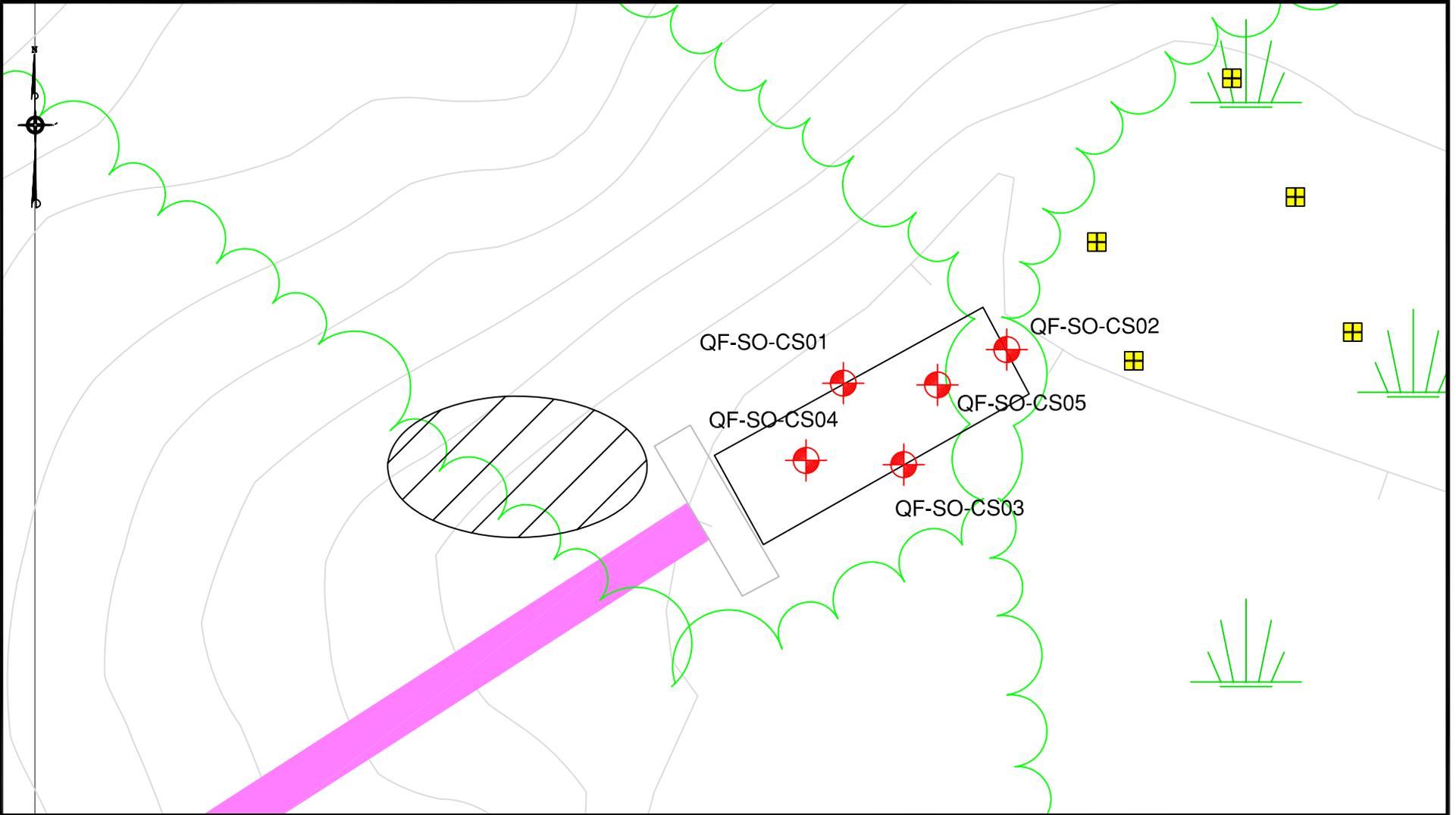
FORMER BUILDING 224 DRAIN LINE AND QDC OUTFALL LOCATION
 CED AREA/QDC OUTFALL 001
 FORMER NCBC DAVISVILLE
 NORTH KINGSTOWN, RHODE ISLAND

SCALE AS NOTED

FILE
\\.\OUTFALL_LOC_MAP.DWG

REV	DATE
0	08/19/10

FIGURE NUMBER
10-3

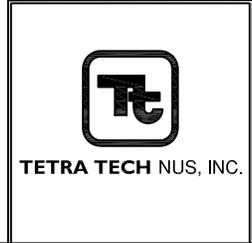
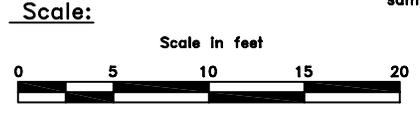


Legend:

 - Limits of former soil stockpile

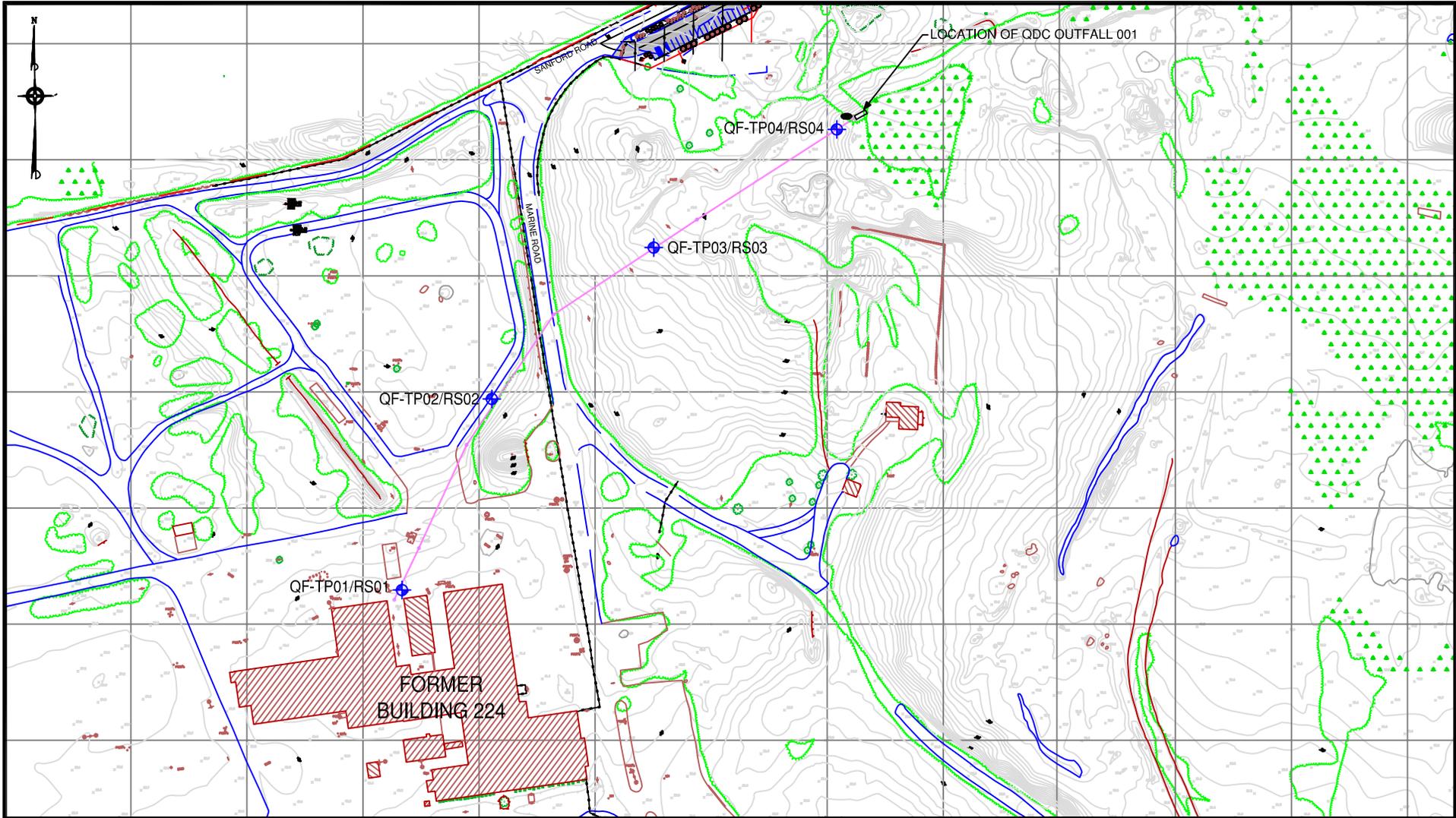
 - Proposed composite soil sample location

 - Proposed sediment sample location



PROPOSED SOIL/SEDIMENT SAMPLING LOCATIONS
 CED AREA/QDC OUTFALL 001
 FORMER NCBC DAVISVILLE
 NORTH KINGSTOWN, RHODE ISLAND

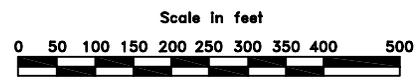
SCALE AS NOTED	
FILE \\...OUTFALL_PROP_SOIL&SED_LOCS.DWG	
REV 0	DATE 08/19/10
FIGURE NUMBER 17-1	



Legend:

- Limits of soil stockpile
- Potential Test Pitting Location

Scale:



TETRA TECH NUS, INC.

PROPOSED TEST PITTING AND SAMPLING LOCATIONS
CED AREA/QDC OUTFALL 001
FORMER NCBC DAVISVILLE
NORTH KINGSTOWN, RHODE ISLAND

SCALE AS NOTED	
FILE \\.\OUTFALL_PROP_TP_LOC.DWG	
REV 0	DATE 08/19/10
FIGURE NUMBER 17-2	

REFERENCES

REFERENCES

Buchman, M. F., 2008. NOAA Screening Quick Reference Tables, NOAA OR&R Report 08-1, Seattle, WA, Office of Response and Restoration Division, National Oceanic and Atmospheric Administration. <http://response.restoration.noaa.gov/cpr/sediment/squirt/squirt.html>

Efroymsen, R.A., M. E. Will, and G. W. Suter. 1997a. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Soil Litter Invertebrates and Heterotrophic Processes, 1997 Revision, ES/ER/TM-126-R2, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Efroymsen, R.A., M. E. Will, G. W. Suter, and A.C. Wooten. 1997b. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants, 1997 Revision, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

United States Environmental Protection Agency (USEPA), 2002. *Guidance for Quality Assurance Project Plans*. EPA QA/G-5, EPA/240/R-02/009. USEPA Office of Environmental Information, Washington DC. December.

USEPA, 2003. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, Attachment B, "Region 1 Tiered Organic and Inorganic Data Validation Guidelines", July 1, 1993, Draft.

USEPA, 2005. *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)*.

United States Environmental Protection Agency Regions 3, 6, and 9. May 2010. Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

APPENDIX A
SITE PHOTOGRAPHS



Date: 11/12/08 Picture No. 1 Location: QDC Outfall 001
Comment: Stone headwall and outfall pipe.



Date: 11/12/08 Picture No. 2 Location: QDC Outfall 001
Comment: Sign identifying QDC Outfall 001.



Date: 11/12/08 Picture No. 3 Location: QDC Outfall 001
Comment: Soil stockpile. Note sign from Picture 2 on right.



Date: 11/12/08 Picture No. 4 Location: QDC Outfall 001
Comment: View looking into outfall pipe.

APPENDIX B

SOIL STOCKPILE SAMPLE ANALYTICAL DATA

RI Analytical Laboratories, Inc.

ONLINE REPORTING SYSTEM

Work Order #: 081222762
Description #: PROJECT# GR08002 TTNUS/FORMER
Date Received: 12/17/2008
Date Completed: 12/18/2008

Global Remediation Services
 1 Westinghouse Plaza Boston, MA 02137

SAMPLE #: 001
SAMPLE DESCRIPTION: OUTFALL SOIL
SAMPLE TYPE: COMPOSITE
SAMPLE DATE/TIME: 12/16/2008 @ 12:00AM

Parameter	Sample Results	Detection Limit	Units	Method	Date Analyzed
Flashpoint	>200	80	deg F	SW846 1010	12/18/2008
Total Petroleum Hydrocarbons					12/18/2008
C6-C10	<75	75	mg/kg dry	SW846 8015B	12/18/2008
C10-C28	10000	25	mg/kg dry	SW846 8015B	12/18/2008
C28-C36	1600	130	mg/kg dry	SW846 8015B	12/18/2008
Extraction date	Extracted			SW846 3545	12/17/2008
PCB					12/18/2008
Aroclor-1016	0.2	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1221	<0.1	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1232	<0.1	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1242	<0.1	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1248	<0.1	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1254	<0.1	0.1	mg/kg dry	SW-846 8082	12/18/2008
Aroclor-1260	0.3	0.1	mg/kg dry	SW-846 8082	12/18/2008
Surrogate			RANGE	SW-846 8082	12/18/2008
Tetrachloro-m-xylene (TCMX)	109		30-150%	SW-846 8082	12/18/2008
Decachlorobiphenyl	35		30-150%	SW-846 8082	12/18/2008
Extraction date	Extracted			SW846 3545	12/17/2008
Volatile Organic Compounds					12/18/2008
Benzene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Bromobenzene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Bromochloromethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Bromodichloromethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Bromoform	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Bromomethane	<0.44	0.44	mg/kg dry	5035/8260B	12/18/2008
n-Butylbenzene	0.79	0.07	mg/kg dry	5035/8260B	12/18/2008

Sec-butylbenzene	0.61	0.07	mg/kg dry	5035/8260B	12/18/2008
tert-Butylbenzene	0.14	0.07	mg/kg dry	5035/8260B	12/18/2008
Carbon Tetrachloride	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Chlorobenzene	2.5	0.07	mg/kg dry	5035/8260B	12/18/2008
Chloroethane	<0.37	0.37	mg/kg dry	5035/8260B	12/18/2008
Chloroform	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Chloromethane	<0.37	0.37	mg/kg dry	5035/8260B	12/18/2008
2-Chlorotoluene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
4-Chlorotoluene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Dibromochloromethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2-Dibromo-3-Chloropropane	<0.15	0.15	mg/kg dry	5035/8260B	12/18/2008
1,2-Dibromoethane(EDB)	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Dibromomethane	<0.15	0.15	mg/kg dry	5035/8260B	12/18/2008
1,2-Dichlorobenzene	0.18	0.07	mg/kg dry	5035/8260B	12/18/2008
1,3-Dichlorobenzene	0.14	0.07	mg/kg dry	5035/8260B	12/18/2008
1,4-Dichlorobenzene	0.95	0.07	mg/kg dry	5035/8260B	12/18/2008
Dichlorodifluoromethane	<0.37	0.37	mg/kg dry	5035/8260B	12/18/2008
1,1-Dichloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2-Dichloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1-Dichloroethene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
cis-1,2-Dichloroethene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
trans-1,2-Dichloroethylene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2-Dichloropropane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,3-Dichloropropane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
2,2-Dichloropropane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1-Dichloropropene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Ethylbenzene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Hexachlorobutadiene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Isopropylbenzene	0.09	0.07	mg/kg dry	5035/8260B	12/18/2008
p-Isopropyltoluene	1.4	0.07	mg/kg dry	5035/8260B	12/18/2008

Methylene Chloride	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
n-Propylbenzene	0.27	0.07	mg/kg dry	5035/8260B	12/18/2008
Naphthalene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Styrene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1,1,2-Tetrachloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1,2,2-Tetrachloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Tetrachloroethene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Toluene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2,3-Trichlorobenzene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2,4-Trichlorobenzene	0.18	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1,1-Trichloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,1,2-Trichloroethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Trichloroethene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Trichlorofluoromethane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2,3-Trichloropropane	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
1,2,4-Trimethylbenzene	1.2	0.07	mg/kg dry	5035/8260B	12/18/2008
1,3,5-Trimethylbenzene	2.0	0.07	mg/kg dry	5035/8260B	12/18/2008
Vinyl Chloride	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
o-Xylene	0.15	0.07	mg/kg dry	5035/8260B	12/18/2008
m,p-Xylene	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Total Xylene	0.15	0.07	mg/kg dry	5035/8260B	12/18/2008
Methyl Tertiary Butyl Ether (MTBE)	<0.07	0.07	mg/kg dry	5035/8260B	12/18/2008
Surrogates			RANGE	5035/8260B	12/18/2008
Dibromofluoromethane	94		70-130%	5035/8260B	12/18/2008
Toluene-d8	101		70-130%	5035/8260B	12/18/2008
4-Bromofluorobenzene	112		70-130%	5035/8260B	12/18/2008
1,2 Dichloroethane-d4	97		70-130%	5035/8260B	12/18/2008
PAH					12/18/2008
Naphthalene	<2.1	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Acenaphthylene	<2.1	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Acenaphthene	6.3	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Fluorene	9.0	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Phenanthrene	24	2.1	mg/kg dry	SW-846 8270D	12/18/2008

Anthracene	8.6	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Fluoranthene	48	8.3	mg/kg dry	SW-846 8270D	12/18/2008
Pyrene	32	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Benzo(a)anthracene	17	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Chrysene	20	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Benzo(b)fluoranthene	26	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Benzo(k)fluoranthene	20	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Benzo(a)pyrene	16	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Indeno(1,2,3-cd)pyrene	6.9	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Dibenzo(a,h)anthracene	3.1	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Benzo(g,h,i)perylene	5.4	2.1	mg/kg dry	SW-846 8270D	12/18/2008
2-Methylnaphthalene	8.3	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Dibenzofuran	<2.1	2.1	mg/kg dry	SW-846 8270D	12/18/2008
Moisture	20		%	SM2540 G.	12/17/2008
Surrogates			RANGE	SW-846 8270D	12/18/2008
Nitrobenzene-d5	127		30-130%	SW-846 8270D	12/18/2008
2-Fluorobiphenyl	130		30-130%	SW-846 8270D	12/18/2008
P-Terphenyl-d14	43		30-130%	SW-846 8270D	12/18/2008
Extraction date	Extracted			SW846 3545	12/17/2008
Arsenic	4.5	1.9	mg/kg dry	SW-846 6010	12/18/2008
Barium	100	0.64	mg/kg dry	SW-846 6010	12/18/2008
Cadmium	3.0	0.32	mg/kg dry	SW-846 6010	12/18/2008
Chromium	48	1.9	mg/kg dry	SW-846 6010	12/18/2008
Lead	420	2.6	mg/kg dry	SW-846 6010	12/18/2008
Mercury	0.17	0.15	mg/kg dry	SW-846 7471A	12/18/2008
Selenium	<13	13	mg/kg dry	SW-846 6010	12/18/2008
Silver	<1.9	1.9	mg/kg dry	SW-846 6010	12/18/2008
Percent Solids	80.0		%	SM2540 G.	12/17/2008
TCLP Metals					12/18/2008
Arsenic	<1.0	1.0	mg/l	SW-846 6010	12/18/2008
Barium	<2.0	2.0	mg/l	SW-846 6010	12/18/2008
Cadmium	0.066	0.050	mg/l	SW-846 6010	12/18/2008
Chromium	<0.30	0.30	mg/l	SW-846 6010	12/18/2008
Lead	1.1	0.40	mg/l	SW-846 6010	12/18/2008
Mercury	<0.010 *	0.010	mg/l	SW-846 7470A	12/18/2008
Selenium	<1.0	1.0	mg/l	SW-846 6010	12/18/2008
Silver	<0.20	0.20	mg/l	SW-846 6010	12/18/2008
Water hg Prep	Digested			SW-846 7470A	12/18/2008

ONLINE REPORTING DISCLAIMER:

Any data obtained from R.I. Analytical Inc.'s Online Data Reporting System is provided by R.I. Analytical Laboratories Inc. solely for the use of the client identified in the report. This data is reported from a databank that may not be finalized and as such, the data should be considered 'draft' and is subject to change. Final data reports are issued from R.I. Analytical Laboratories as approved Certificates of Analysis. The format of the data as presented in this Online Data Reporting System does not meet NELAC reporting criteria.

APPENDIX C
FIELD DOCUMENTATION FORMS



TETRA TECH NUS, INC.

PHOTOIONIZATION DETECTOR FIELD CALIBRATION LOG

Serial No.: _____

Model No.: _____

Decal No.: _____

Site Name/Location: _____

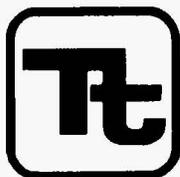
Tetra Tech NUS Charge No.: _____

CALIBRATION DATE	STANDARD GAS- ISOBUTYLENE	(AM) CALIBRATION READING Isobutylene Equiv. (ppm)	(PM) CALIBRATION CHECK Isobutylene Equiv. (ppm)	SIGNATURE	COMMENTS
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	
	Lot # _____ Conc. = _____ ppm	/		AM: PM:	



PROJECT NO:		FACILITY:		PROJECT MANAGER		PHONE NUMBER		LABORATORY NAME AND CONTACT:							
SAMPLERS (SIGNATURE)				FIELD OPERATIONS LEADER		PHONE NUMBER		ADDRESS							
				CARRIER/WAYBILL NUMBER				CITY, STATE							
STANDARD TAT <input type="checkbox"/> RUSH TAT <input type="checkbox"/> <input type="checkbox"/> 24 hr. <input type="checkbox"/> 48 hr. <input type="checkbox"/> 72 hr. <input type="checkbox"/> 7 day <input type="checkbox"/> 14 day				TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAB (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G)						
PRESERVATIVE USED															
DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TYPE OF ANALYSIS				COMMENTS							
1. RELINQUISHED BY				DATE		TIME		1. RECEIVED BY				DATE		TIME	
2. RELINQUISHED BY				DATE		TIME		2. RECEIVED BY				DATE		TIME	
3. RELINQUISHED BY				DATE		TIME		3. RECEIVED BY				DATE		TIME	
COMMENTS															

APPENDIX D
SAMPLING AND RELATED SOPs



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number CT-05	Page 1 of 7
Effective Date 01/29/01	Revision 2
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.

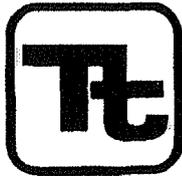
ATTACHMENT A



MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: _____ CTO: _____ Project Manager: _____ Requestor: _____ Program/Client: _____ State/EPA Region: _____	Request Date: _____ Date Data Available for Production: _____ Request in Support of: _____ Database Lead: _____ GIS Lead: _____ 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: _____	
Labels: <input type="checkbox"/> Labels needed for an upcoming sampling event _____ Total # of Samples _____ Estimated Hours Additional Instructions: _____ _____ Due Date _____ Complete ETS Charge No. _____ _____ FOL _____	
Data Entry: <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. _____	
Tables: <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. _____	
GIS: <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. _____	
Statistics: <input type="checkbox"/> Yes _____ Estimated Hours Additional Instructions: _____ _____ Due Date _____ Complete ETS Charge No. _____	
Geostatistics: <input type="checkbox"/> Yes _____ Estimated Hours Additional Instructions: _____ _____ Due Date _____ Complete ETS Charge No. _____	



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	GH-1.5	Page	1 of 20
Effective Date	06/99	Revision	1
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</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.

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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 Φ -1/2 inch Φ)" 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:

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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

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
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 (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**

Particle Name	Grain Size Diameter
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 ₂ 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 Drilling Area
 2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z Background (ppm):

NIX CORE IN BEDROCK RUN (1) = 25 min, RUN (2) = 15 min

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

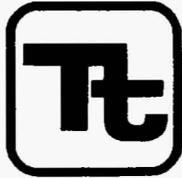
Unified Soil Classification System (USCS).

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	12/03	Revision	2
Applicability	Tetra Tech NUS, Inc.		
Prepared	Health & Safety		
Approved	D. Senovich <i>DS</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**

<p>Alabama Alabama One-Call 1-800-292-8525</p> <p>Alaska Locate Call Center of Alaska, Inc. 1-800-478-3121</p> <p>Arizona Arizona Blue Stake 1-800-782-5348</p> <p>Arkansas Arkansas One Call System, Inc. 1-800-482-8998</p> <p>California Underground Service Alert North 1-800-227-2600 Underground Service Alert of Southern California 1-800-227-2600</p> <p>Colorado Utility Notification Center of Colorado 1-800-922-1987</p> <p>Connecticut Call Before You Dig 1-800-922-4455</p> <p>Delaware Miss Utility of Delmarva 1-800-282-8555</p> <p>Florida Sunshine State One-Call of Florida, Inc. 1-800-432-4770</p> <p>Georgia Underground Protection Center, Inc. 1-800-282-7411</p> <p>Hawaii Underground Service Alert North 1-800-227-2600</p> <p>Idaho Dig Line Inc. 1-800-342-1585 Kootenai County One-Call 1-800-428-4950 Shoshone - Benewah One-Call 1-800-398-3285</p> <p>Illinois JULIE, Inc. 1-800-892-0123 Digger (Chicago Utility Alert Network) 312-744-7000</p> <p>Indiana Indiana Underground Plant Protection Service 1-800-382-5544</p>	<p>Iowa Iowa One-Call 1-800-292-8989</p> <p>Kansas Kansas One-Call System, Inc. 1-800-344-7233</p> <p>Kentucky Kentucky Underground Protection Inc. 1-800-752-6007</p> <p>Louisiana Louisiana One Call System, Inc. 1-800-272-3020</p> <p>Maine Dig Safe System, Inc. 1-888-344-7233</p> <p>Maryland Miss Utility 1-800-257-7777 Miss Utility of Delmarva 1-800-282-8555</p> <p>Massachusetts Dig Safe System, Inc. 1-888-344-7233</p> <p>Michigan Miss Dig System, Inc. 1-800-482-7171</p> <p>Minnesota Gopher State One Call 1-800-252-1168</p> <p>Mississippi Mississippi One-Call System, Inc. 1-800-227-6477</p> <p>Missouri Missouri One-Call System, Inc. 1-800-344-7483</p> <p>Montana Utilities Underground Protection Center 1-800-424-5555 Montana One Call Center 1-800-551-8344</p> <p>Nebraska Diggers Hotline of Nebraska 1-800-331-5686</p> <p>Nevada Underground Service Alert North 1-800-227-2600</p> <p>New Hampshire Dig Safe System, Inc. 1-888-344-7233</p>	<p>New Jersey New Jersey One Call 1-800-272-1000</p> <p>New Mexico New Mexico One Call System, Inc. 1-800-321-2537 Las Cruces- Dona Ana Blue Stakes 1-888-526-0400</p> <p>New York Dig Safely New York 1-800-962-7962 New York City- Long Island One Call Center 1-800-272-4480</p> <p>North Carolina The North Carolina One-Call Center, Inc. 1-800-632-4949</p> <p>North Dakota North Dakota One-Call 1-800-795-0555</p> <p>Ohio Ohio Utilities Protection Service 1-800-362-2764 Oil & Gas Producers Underground Protect'n Svc 1-800-925-0988</p> <p>Oklahoma Call Okie 1-800-522-6543</p> <p>Oregon Oregon Utility Notification Center/One Call Concepts 1-800-332-2344</p> <p>Pennsylvania Pennsylvania One Call System, Inc. 1-800-242-1776</p> <p>Rhode Island Dig Safe System, Inc. 1-888-344-7233</p> <p>South Carolina Palmetto Utility Protection Service Inc. 1-888-721-7877</p> <p>South Dakota South Dakota One Call 1-800-781-7474</p> <p>Tennessee Tennessee One-Call System, Inc. 1-800-351-1111</p>
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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

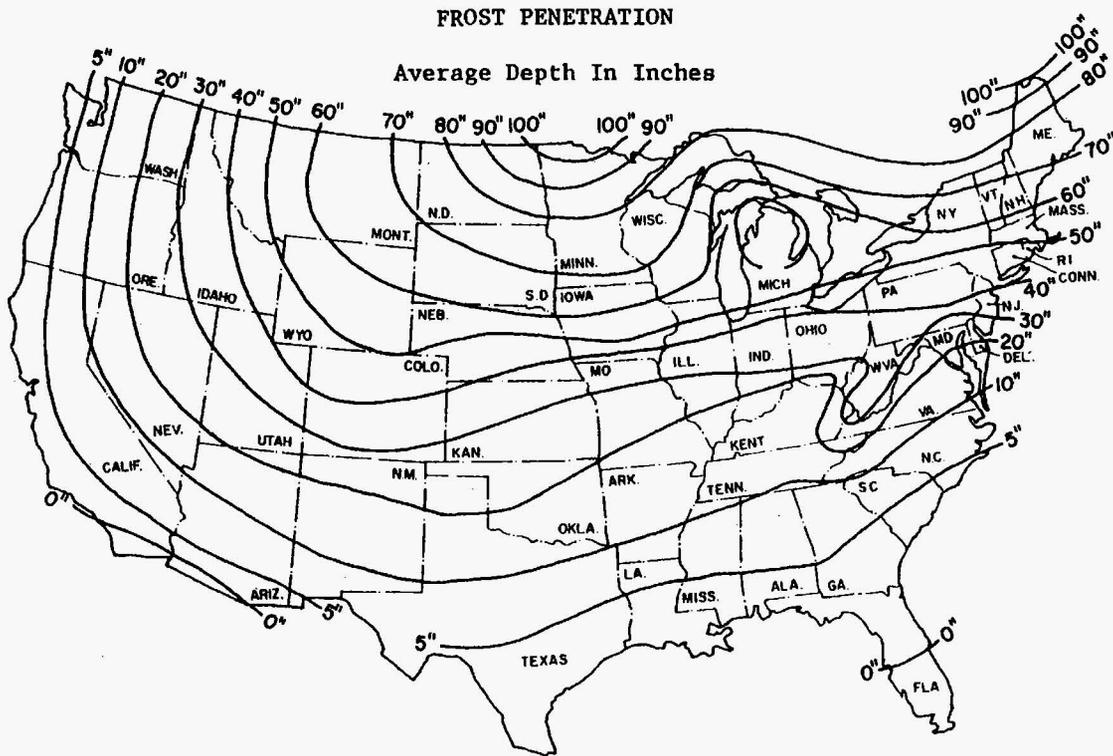
Ontario One-Call System
1-800-400-2255

Quebec

Info-Excavation
1-800-663-9228

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

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TETRA TECH

STANDARD OPERATING PROCEDURES

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Effective Date	03/28/2008	Revision	5
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
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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® 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® 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.

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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

OBSERVATIONS / NOTES:	MAP:

Circle if Applicable:		Signature(s):
<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.



TETRA TECH

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

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clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket.**

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.

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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 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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. 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.

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~~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.~~

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.

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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.
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.

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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:

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:

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- 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.

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.

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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.
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.

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- 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.

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)

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- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags
- 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:

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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.
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.).

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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.
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

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hazards such 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).
- 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.

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- 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.
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,~~

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~~responsibilities, and procedures are described in SOP SA-2.5.~~

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:

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- The purpose and extent of the exploration
- 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

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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, 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.

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6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- 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 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.

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- 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.)
- 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.

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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.

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.~~

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- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

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

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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.

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/O₂ 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:

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- Calibration logs
- Excavation inspection checklists
- 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.

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USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

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**ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET**



Tetra Tech NUS, Inc.

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):				

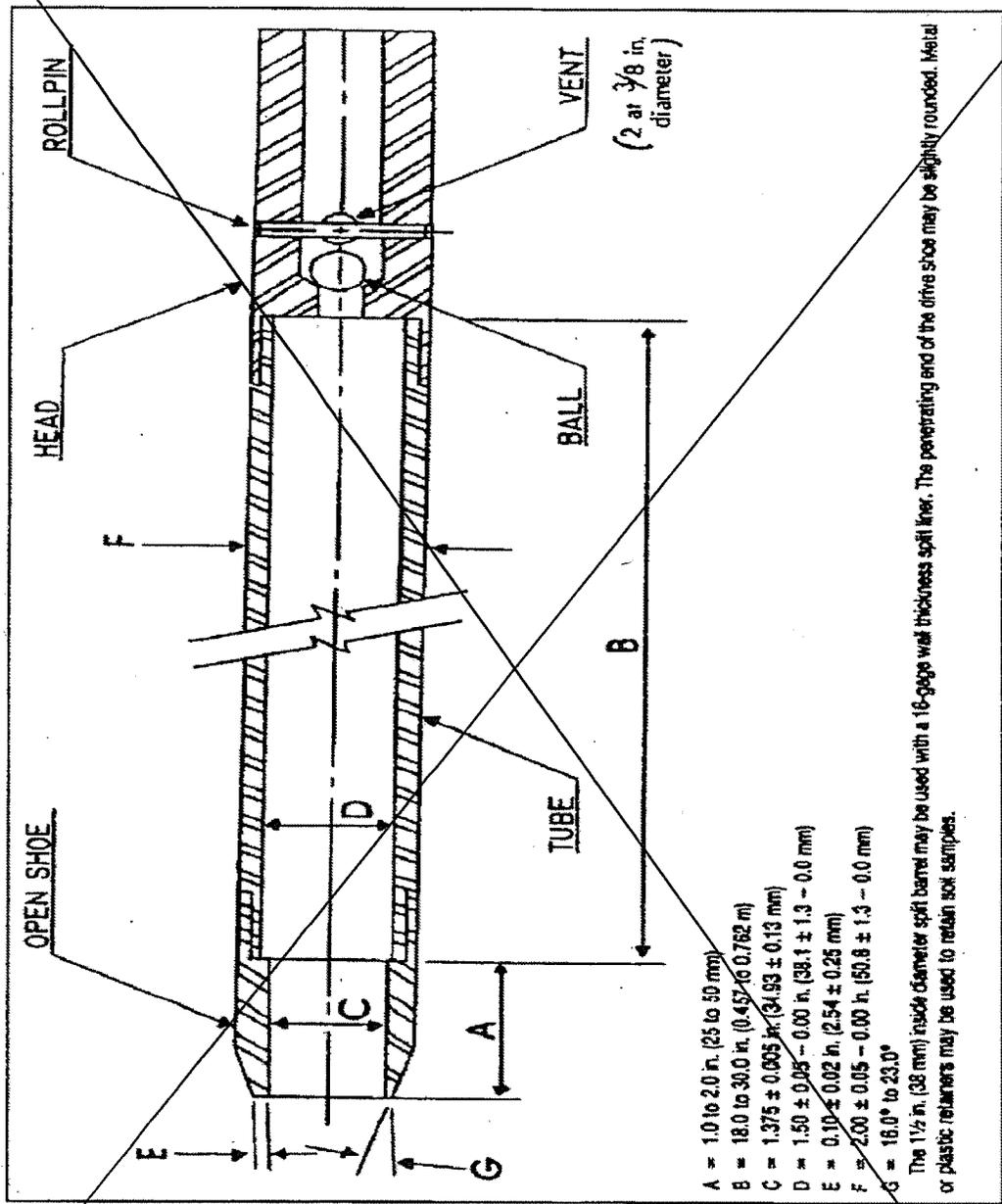
SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:	MAP:

Circle if Applicable:	Signature(s):
MS/MSD Duplicate ID No.:	

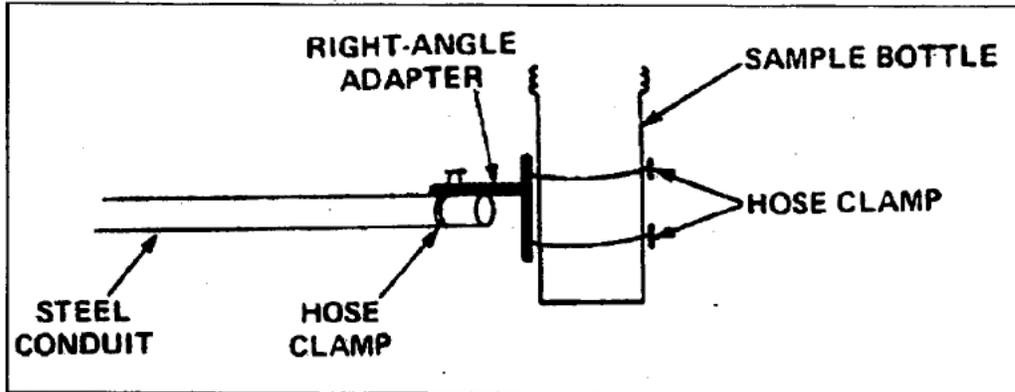
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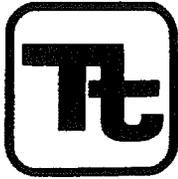
**ATTACHMENT B
SPLIT-SPOON SAMPLER**



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**ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING**





TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Effective Date	02/04	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject
NON-RADIOLOGICAL SAMPLE HANDLING

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H₂SO₄
- Nitric Acid - HNO₃
- Sodium Hydroxide - NaOH

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Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na₂S₂O₃

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

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changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO₃, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H ₂ SO ₄)	1 part concentrated H ₂ SO ₄ : 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO ₃)	Undiluted concentrated HNO ₃	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

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- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.
- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).
- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

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- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.
- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)
- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.
- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.
- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.
- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).
- Seal (i.e., tape or tie top in knot) large liner bag.
- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.
- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

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Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

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ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration	Container ⁽¹⁾	Sample Size	Preservation ⁽²⁾	Holding Time ⁽²⁾
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WATER

Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days ⁽⁹⁾
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO ₃ to pH ≤ 2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days

SOIL

Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorga nic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction

AIR

Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended
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1 All glass containers should have Teflon cap liners or septa.

2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

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ATTACHMENT B

**ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES**

Parameter Number/Name	Container ⁽¹⁾	Preservation ⁽²⁾⁽³⁾	Maximum Holding Time ⁽⁴⁾
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INORGANIC TESTS:

Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid ⁽⁵⁾	14 days ⁽⁶⁾
Fluoride	P	None required	28 days
Hardness	P, G	HNO ₃ to pH 2; H ₂ SO ₄ to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H ₂ SO ₄ to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

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**ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES
PAGE TWO**

Parameter Number/Name	Container ⁽¹⁾	Preservation ⁽²⁾⁽³⁾	Maximum Holding Time ⁽⁴⁾
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INORGANIC TESTS (Cont'd):

Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours

METALS:⁽⁷⁾

Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO ₃ to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO ₃ to pH 2	6 months

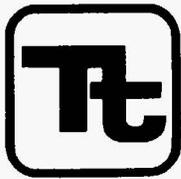
ORGANIC TESTS:⁽⁸⁾

Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ HCl to pH 2 ⁽⁹⁾	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ adjust pH to 4-5 ⁽¹⁰⁾	14 days
Phenols ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
Benzidines ^{(11), (12)}	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction ⁽¹³⁾
Phthalate esters ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines ^{(11), (14)}	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
PCBs ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ ; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs) ^{(11), (14)}	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ ; store in dark	7 days until extraction; 40 days after extraction
Haloethers ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF) ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction

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**ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES
PAGE THREE**

- (1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
- (4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- (7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- (9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
- (10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- (11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
- (12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
- (13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- (14) For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- (15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Effective Date 09/03	Revision 2
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>ds</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 field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS 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

Project Manager (PM) - The Project Manager 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 Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

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 onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

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that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS 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, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered 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, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed 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 separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

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5.3 **Field Forms**

All Tetra Tech NUS 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 contingent upon 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 SOP.

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. A log sheet must be completed 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. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) 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 for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while 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. It 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. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

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5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used 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

A Groundwater Level Measurement Sheet must be filled out 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. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed 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

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is collected, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool 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 assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, 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. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite 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 useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 **Daily Activities Report**

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

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5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which 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 Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at <http://intranet.ttnus.com> under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. [HTTP://INTRANET.TTNUS.COM](http://intranet.ttnus.com) CLICK ON FIELD LOG SHEETS

- Groundwater Sample Log Sheet
- Surface Water Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Container Sample and Inspection Sheet
- Geochemical Parameters (Natural Attenuation)
- Groundwater Level Measurement Sheet
- Pumping Test Data Sheet
- Packer Test Report Form
- Boring Log
- Monitoring Well Construction Bedrock Flush Mount
- Monitoring Well Construction Bedrock Open Hole
- Monitoring Well Construction Bedrock Stick Up
- Monitoring Well Construction Confining Layer
- Monitoring Well Construction Overburden Flush Mount
- Monitoring Well Construction Overburden Stick Up
- Test Pit Log
- Monitoring Well Materials Certificate of Conformance
- Monitoring Well Development Record

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Daily Activities Record
 Field Task Modification Request
 Hydraulic Conductivity Test Data Sheet
 Low Flow Purge Data Sheet
 QA Sample Log Sheet
 Equipment Calibration Log
 Field Project Daily Activities Checklist
 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 manger 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

	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:	



TETRA TECH NUS, INC.

CHAIN OF CUSTODY

NUMBER 3413

PAGE ___ OF ___

PROJECT NO:		FACILITY:		PROJECT MANAGER		PHONE NUMBER		LABORATORY NAME AND CONTACT:							
SAMPLERS (SIGNATURE)				FIELD OPERATIONS LEADER		PHONE NUMBER		ADDRESS							
				CARRIER/WAYBILL NUMBER				CITY, STATE							
STANDARD TAT <input type="checkbox"/> RUSH TAT <input type="checkbox"/> <input type="checkbox"/> 24 hr. <input type="checkbox"/> 48 hr. <input type="checkbox"/> 72 hr. <input type="checkbox"/> 7 day <input type="checkbox"/> 14 day				TOP DEPTH (FT)		BOTTOM DEPTH (FT)		MATRIX (GW, SO, SW, SD, QC, ETC.)		COLLECTION METHOD GRAP (G) COMP (C)		No. OF CONTAINERS		CONTAINER TYPE PLASTIC (P) or GLASS (G)	
DATE YEAR		TIME												SAMPLE ID	
1. RELINQUISHED BY				DATE		TIME		1. RECEIVED BY		DATE		TIME			
2. RELINQUISHED BY				DATE		TIME		2. RECEIVED BY		DATE		TIME			
3. RELINQUISHED BY				DATE		TIME		3. RECEIVED BY		DATE		TIME			
COMMENTS															

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE)

YELLOW (FIELD COPY)

PINK (FILE COPY)

4/02R
FORM NO. TINUS-001

ATTACHMENT C

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ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

Signature <hr/> Date <hr/> CUSTODY SEAL		CUSTODY SEAL <hr/> Date <hr/> Signature
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STANDARD OPERATING PROCEDURES

Number SA-7.1	Page 1 of 16
Effective Date 03/28/2008	Revision 4
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 DW Label

INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE _____ JOB NO. _____

LOCATION _____

DATE _____

DRUM# _____

CONTENTS _____

VOLUME _____

CONTACT _____

EMERGENCY PHONE NUMBER _____

APPENDIX E
ANALYTICAL SPECIFICATION

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

**REVISED TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
CED AREA/QDC OUTFALL 001
FORMER NAVAL CONSTRUCTION BATTALION CENTER (NCBC) DAVISVILLE
NORTH KINGSTOWN, RHODE ISLAND**

**COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62472-03-D-0057, CONTRACT TASK ORDER (CTO) NO. 19**

**CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
CHEMICAL ANALYSIS**

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62472-03-D-0057, is procuring laboratory analytical services to support confirmatory sampling and a drain line investigation at QDC Outfall 001 at NCBC Davisville, North Kingstown, Rhode Island. Requested analyses include volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), target analyte list (TAL) metals, and total petroleum hydrocarbons (TPH) as gasoline range organics (GRO) ranges C₅ – C₁₂ and extractable petroleum hydrocarbons (ExTPH) ranges C₉-C₄₀.

The laboratory performing these analyses must be certified by the State of Rhode Island. Additionally, the laboratory must provide a copy of the Department of Defense (DOD) Environmental Laboratory Accreditation Program (ELAP) accreditation letter; all methods and target analytes requested must be addressed.

The responding laboratory may be required to submit detection limits (DLs), limits of detection (LODs) and limits of quantitation (LOQs) for all analyses and matrices requested. After award, the laboratory may be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory may also be asked to complete tables for inclusion in the Sampling and Analysis Plan (SAP). The SAP will be prepared according to the Uniform Federal Policy (UFP) for QAPPs (March 2005) and utilize the UFP SAP worksheets 1 through 37.

2.0 SAMPLE INFORMATION

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in Table 1. This investigation includes analysis of soil, sediment, and residual material samples.

The sampling is scheduled for the first quarter of 2011. The exact date of sample collection will be communicated to the laboratory at least seven days in advance.

The samples are expected to be of low or moderate contaminant concentration. The field crew will attempt to identify any potentially high concentration samples.

Field duplicate samples will be submitted to the laboratory with "blinded" identification. The field crew will designate samples (one per twenty samples of like matrices) for matrix spike/matrix spike duplicate (MS/MSD) (organic) and matrix spike/laboratory duplicate (metal) analyses. Additional volumes of these samples will be provided as necessary.

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The residual material samples will be collected from inside a drainage pipe and are expected to be similar to sediment samples. For the non-volatile soil, sediment, and residual material analyses (SVOC, PAH, PCB, ExTPH, and metals), the laboratory must decant any standing water, homogenize the sample, and **determine the percent moisture before sample analysis**. The laboratory must increase the sample aliquot to compensate for the moisture content of the samples. The project screening levels (PSLs) listed in Revised Attachment A must be met, to the extent technically possible using the requested methods, regardless of the moisture content of the soil, sediment, and residual material samples.

If the percent moisture content is too high and the soil, sediment, or residual material sample contains noticeable organic material, further dewatering must be performed at the laboratory prior to non-volatile sample analysis. This could be accomplished by freeze-drying under controlled conditions proven to recover the analysis-specific surrogates; centrifugation and decanting free water; low temperature oven drying (below 60°C); or other procedure proposed by the laboratory and approved by TtNUS.

VOC and GRO Analysis

Soil, sediment, and residual material samples for VOC and GRO analysis will be collected using a coring device (cut off syringe). The following aliquots will be collected:

- Three-40 ml VOC vials pre-preserved with 5 ml of methanol (one for VOCs, two for GRO)
- Two-40 ml VOC vials pre-preserved with 5 ml of VOC-free reagent water w/ a magnetic stir bar
- One-2 oz wide mouth jar for percent solids

The pre-preserved VOC and GRO sample containers must be weighed accurately to within 0.01 grams and identified with a unique ID number. Both the ID number and the applicable vial weight must be recorded on a weight tracking form for return shipment to the laboratory. When samples are received at the laboratory, the pre-preserved vials must be re-weighed and these values recorded in the weight tracking form. TtNUS must be contacted immediately if leaking vials are received at the laboratory.

The VOC low concentration analysis (water preserved vial) must be performed first for all the samples in order to meet the PSLs. The methanol-preserved vials should be used to perform a second analysis at a dilution if needed.

The reagent water pre-preserved VOC vials **MUST** be analyzed within 48 hours from collection, or they may be frozen at < -10°C and analyzed within 14 days of sample collection.

GRO/ExTPH Analyses

The GRO fraction must include all petroleum hydrocarbons ranging from C₅ to C₁₂. The ExTPH analysis must be extended to include the C₉ to C₄₀ petroleum hydrocarbons. The full range of petroleum products up to C₄₀ must be quantitated. The laboratory must perform a quantitative analysis and also identify the types of fuel contamination present. The gas chromatograms must depict the finger print patterns at greater than 25 percent of the full scale.

SVOC and PAH Analyses

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SVOC full scan analysis is specified for the list of SVOCs in Revised Attachment A, and SIM analysis is specified for PAHs. If any PAH concentration analyzed by SIM exceeds the upper instrument calibration level and an acceptable result is obtained from full scan analysis, there is no need to perform dilutions for the SIM analysis; the result may be reported from the full scan analysis.

PCB Analysis

All positive identifications for PCBs by gas chromatography (GC) with electron capture detector (ECD) MUST be confirmed on a second column that possesses retention characteristics different from those exhibited by the first column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the laboratory LOQ but greater than the DL must be reported by the laboratory; the laboratory must "J" flag these results. The laboratory should designate and always use the same chromatographic stationary phase for Column 1 and likewise for Column 2. Results should be reported from Column 1. However, if the between-column RPD exceeds 40%, the analyst must select which result (i.e., from Column 1 or Column 2) to report; and the laboratory must provide an explanation in the case narrative why the particular result was selected for each affected target analyte.

Metal Analysis

For the ICP and mercury metal analyses, the laboratory must perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. All metals must be spiked and matrix spike recoveries for all metals must be reported. If the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (serial dilution) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

3.0 ANALYSIS/REPORTING INFORMATION

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables (EDDs) to be provided in the format described in Attachment C. The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Analysis and reporting requirements addressed in the DOD Quality Systems Manual (QSM) (March 2009) and the requested methods must be followed. **All organic and metal analyses must include analysis of a second-source initial calibration verification (ICV) sample as required by the QSM.** For GRO, the LCS in each batch may serve as the ICV if it is a second source, non-prepared standard. The laboratory-derived recovery limits for matrix spike, LCSs, and surrogates must be met. Additionally, it is a requirement of TtNUS that the associated hard copy/PDF data packages for the VOC, SVOC, PAH, PCB, and metals analyses must meet Contract Laboratory Program (CLP) format, reporting, and PDF/hard copy data package deliverable requirements. However, the CLP format must be modified to present the sample-specific DLs, LODs, and LOQs for each analyte on the Form I. Also, the initial calibration verification results must be reported on a summary form. The PDF and hard copy data packages for the remaining analyses must be in a CLP-modified format and must include the appropriate

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summary forms and raw data for all samples and laboratory quality control samples. The summary forms should include the method-specific quality control limits (recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.). The information in the header of the forms must be complete. The TtNUS sample identification numbers **must be included** on the raw data and summary forms.

Additionally, each hard copy and PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP, Forms 1 through 15; for non-CLP it shall be the CLP-like equivalent of Forms 1 through 15).

Revised Attachment A details the required target compound list and required PSLs to be achieved by the laboratory.

Sample analytical results for VOCs, SVOCs, PCBs, and Metals must be reported down to the DL, with non-detected results qualified as "U" and reported with an associated value of the LOD; positive results between the LOQ and DL must be qualified as "J". Sample results for GRO, ExTPH, and PAHs must be reported down to the LOQ.. Soil, sediment, and residual material samples must be reported on a dry-weight basis.

The hard copy/PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported in the raw data and Form Is for organic analyses. For inorganic analyses, only the final sample results should be reported in the Form Is, and data from all analytical runs must be included in the raw data.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of sample collection. The holding times are as follows:

Analyses	Preservation	Holding Time
VOCs	Solid: 5 ml VOC-free reagent water; cool to 4° C and freeze at < -10° C within 48 hours	14 days to analysis
	Solid: 5 ml methanol; cool to 4° C	14 days to analysis
GRO	Solid: 5 ml methanol; cool to 4° C	14 days to analysis

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Analyses	Preservation	Holding Time
SVOCs, PAHs, PCBs, ExTPH	Solid: Cool to 4° C	14 days to extraction; 40 days to analysis
Metals	Solid: Cool to 4° C	6 months to analysis for ICP metals; 28 days for mercury

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection for samples preserved as requested in the analytical methods. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, **the holding time criteria apply to any/all subsequent sample dilutions and re-analyses.**

The TtNUS Project Manager for this project is Mr. Stephen Vetere, and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To insure good communication it is required that the laboratory's appointed project manager contact Mr. Vetere once a week for the entire project duration.

Contact information for Mr. Vetere is as follows:

Tetra Tech NUS
 55 Jonspin Road
 Wilmington, MA 01887
 Phone: 978-474-8444
 Fax: 978-474-8499
 Email: stephen.vetere@tetrattech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All hard copy/PDF (2 CDs) analytical data packages and associated electronic (TXT) deliverables are due within a turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG).

The SDGs must contain up to 20 samples. The frequency with which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed.

The hard copy analytical data package, 1 PDF (CD) copy of the analytical data package, **and the original chain-of-custody form** (received with the samples and signed by the laboratory sample custodian) should be sent to Ms. Lucy Guzman. Contact information for Ms. Guzman is the same as noted above for Mr. Vetere except that her direct phone number is (978) 474-8416 and her email address is lucy.guzman@tetrattech.com.

The electronic (TXT) deliverables, 1 PDF (CD) copy of the analytical data package, and **a copy of the chain-of-custody form**, should be sent to Ms. Tobrena Skeen. Contact information for Ms. Skeen is as follows:

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661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220-2745
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.skeen@tetrattech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be picked up by the laboratory's courier at least every other day of sample collection, in coordination with the Tetra Tech field operations leader. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. The laboratory will be notified at least seven days prior to sample collection.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory is to provide all necessary sample containers (**plus approximately 10% extra for breakage**). All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. Each cooler must include a "temperature blank" vial. **The laboratory must also provide any extra coolers needed for return shipment of samples to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

The internal transfers of samples and extracts within the laboratory must be accomplished and documented as controlled custody transfers. The laboratory must maintain and submit documentation that supports an unbroken chain of custody for samples and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact leftover samples, as stipulated in the MSA. **Additionally, the laboratory must store PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated, without prior notification and consent from the CLEAN Subcontracting Officer.

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The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Lucy Guzman at 978-474-8416 or via e-mail lucy.guzman@tetrattech.com. Ms. Guzman must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetrattech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte quantitation limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. All costing information must reflect the terms and conditions established by the 2010 CLEAN MSA.

**TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
CED AREA/QDC OUTFALL 001, NCBC DAVISVILLE
NORTH KINGSTOWN, RHODE ISLAND**

Matrix	Parameter⁽¹⁾	Method	# Samples	Unit Price	Total Cost
Soil	VOCs	SW-846 5035/8260C	13	\$	\$
	SVOCs (full scan)	SW-846 3540C, 3550B, or 3570/8270D	11	\$	\$
	PAHs (SIM)	SW-846 3540C, 3550B, or 3570/8270D SIM	11	\$	\$
	PCBs	SW-846 3540C, 3550B, or 3570/8082A	11	\$	\$
	GRO (C5-C12)	SW-846 5035/8015D	13	\$	\$
	ExTPH (C9-C40)	SW-846 3540C, 3550B, or 3570/8015D	11	\$	\$
	TAL Metals	SW-846 3050B/6010C ⁽²⁾ /6020A ⁽²⁾ /7471B	11	\$	\$
Sediment	VOCs	SW-846 5035/8260C	7	\$	\$
	SVOCs (full scan)	SW-846 3540C, 3550B, or 3570/8270D	6	\$	\$
	PAHs (SIM)	SW-846 3540C, 3550B, or 3570/8270D SIM	6	\$	\$
	PCBs	SW-846 3540C, 3550B, or 3570/8082A	6	\$	\$
	GRO (C5-C12)	SW-846 5035/8015D	7	\$	\$
	ExTPH (C9-C40)	SW-846 3540C, 3550B, or 3570/8015D	6	\$	\$
	TAL Metals	SW-846 3050B/6010C ⁽²⁾ /6020A ⁽²⁾ /7471B	6	\$	\$
Residual Material	VOCs	SW-846 5035/8260C	7	\$	\$
	SVOCs (full scan)	SW-846 3540C, 3550B, or 3570/8270D	6	\$	\$
	PAHs (SIM)	SW-846 3540C, 3550B, or 3570/8270D SIM	6	\$	\$
	PCBs	SW-846 3540C, 3550B, or 3570/8082A	6	\$	\$
	GRO (C5-C12)	SW-846 5035/8015D	7	\$	\$
	ExTPH (C9-C40)	SW-846 3540C, 3550B, or 3570/8015D	6	\$	\$
	TAL Metals	SW-846 3050B/6010C*/6020A*/7471B	6	\$	\$

(1) Required target analytes are presented in Revised Attachment A.

(2) Arsenic, cobalt, silver, and thallium will be analyzed by 6020A; remaining ICP metals will be analyzed by 6010C.

TOTAL \$

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there a charge and what is that charge?_____

Name of Laboratory_____

Signature_____

ATTACHMENT A
Required Target Analyte List and Project Screening Levels
(See attached Word tables)

**ATTACHMENT A
CED AREA/QDC OUTFALL 001, NCBC DAVISVILLE
NORTH KINGSTOWN, RHODE ISLAND
REQUIRED TARGET ANALYTE LIST AND PROJECT SCREENING LEVELS**

SAP Worksheet #15a – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Notes for all matrices: Sample analytical results for VOCs, SVOCs, PCBs, and Metals will be reported down to the Detection Limit (DL), with non-detected results qualified as “U” and reported with an associated value of the Limit of Detection (LOD); positive results between the LOQ and DL will be qualified as “J” (estimated) due to uncertainty below the LOQ. Sample results for GRO, ExTPH, and PAHs will be reported down to the LOQ. Mitkem does not report GRO and ExTPH results below the LOQ due to lack of confirmation column analysis in the method. PAHs will be analyzed in SIM mode; Mitkem does not report SIM results below the LOQ due to limited mass spectral data, which can lead to false positive detections. For the GRO, ExTPH, and PAH analyses, Mitkem will analyze a standard at the LOQ.

Mitkem will analyze for and report individual Aroclors and Tetra Tech will calculate PCBs (Total).

Select metals (arsenic, cobalt, silver, and thallium) will be analyzed by EPA SW-846 Method 6020A, instead of Method 6010C, in order to achieve lower detection limits to meet the PSLs. Although not all of these metals would require the lower detection limits for each matrix, the same metals will be analyzed by Method 6020A across all matrices in order to simplify the sampling and analysis procedures.

In this Worksheet #15, the Project Screening Level (PSL) is presented in bold font if it is less than the LOQ but greater than or equal to the LOD; and the PSL is presented as bolded and shaded if it is less than the LOD. The limitations on data usability due to unmet sensitivity goals will be evaluated as described in Worksheet #37 and will be discussed in the project report.

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Volatile Organic Compounds									
1,1,1-Trichloroethane	71-55-6	8260C	870	29.8	Region 5 ESL	9.9	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	0.56	0.127	Region 5 ESL	0.042	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.1	1.1	EPA RSL Res	0.37	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	3.3	3.3	EPA RSL Res	1.1	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	24	8.28	Region 5 ESL	2.8	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	190	2.96	Region 5 ESL	0.99	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.43	0.43	EPA RSL Res	0.14	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	0.89	0.89	EPA RSL Res	0.3	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	6.2	6.2	EPA RSL Res	2.1	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	6.2	6.2	EPA RSL Res	2.1	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	78	78	EPA RSL Res	26	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	--	37.7	Region 5 ESL	13	0.005	0.002	0.0007
1,4 Dichlorobenzene	106-46-7	8260C	2.4	0.546	Region 5 ESL	0.18	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	2800	89.6	Region 5 ESL	30	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	21	12.6	Region 5 ESL	4.2	0.005	0.004	0.00083

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4-Methyl-2-Pentanone	108-10-1	8260C	530	443	Region 5 ESL	150	0.005	0.004	0.00073
Acetone	67-64-1	8260C	6100	2.5	Region 5 ESL	0.83	0.005	0.004	0.0016
Benzene	71-43-2	8260C	1.1	0.255	Region 5 ESL	0.085	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	0.27	0.27	EPA RSL Res	0.09	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	61	15.9	Region 5 ESL	5.3	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	0.73	0.235	Region 5 ESL	0.078	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	82	0.0941	Region 5 ESL	0.031	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.61	0.61	EPA RSL Res	0.2	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	29	13.1	Region 5 ESL	4.4	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	1500	1500	EPA RSL Res	500	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.29	0.29	EPA RSL Res	0.097	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	12	10.4	Region 5 ESL	3.5	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	78	0.78373	Region 5 ESL	0.26	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	1.7	0.398	Region 5 ESL	0.13	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	0.68	0.68	EPA RSL Res	0.23	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	5.4	5.16	Region 5 ESL	1.7	0.005	0.002	0.0005
Methylene Chloride	75-09-2	8260C	11	4.05	Region 5 ESL	1.4	0.005	0.002	0.0013
Styrene	100-42-5	8260C	630	4.69	Region 5 ESL	1.6	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.55	0.55	EPA RSL Res	0.18	0.005	0.002	0.00062
Toluene	108-88-3	8260C	500	5.45	Region 5 ESL	1.8	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	15	0.784	Region 5 ESL	0.26	0.005	0.002	0.00053
trans-1,3-Dichloropropene	10061-02-6	8260C	1.7	0.398	Region 5 ESL	0.13	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	2.8	2.8	EPA RSL Res	0.93	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	79	16.4	Region 5 ESL	5.5	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	0.06	0.06	EPA RSL Res	0.02	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	63	10	Region 5 ESL	3.3	0.005	0.002	0.00047
Semivolatle Organic Compounds									
2,4,5-Trichlorophenol	95-95-4	8270D	610	4	ORNL Plant	1.3	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	6.1	6.1	EPA RSL Res	2	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	18	18	EPA RSL Res	6	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	120	0.01	Region 5 ESL	0.0033	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	12	0.0609	Region 5 ESL	0.02	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	1.6	1.28	Region 5 ESL	0.43	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	6.1	0.0328	Region 5 ESL	0.011	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	630	0.0122	Region 5 ESL	0.0041	0.33	0.067	0.052
2-Chlorophenol	95-57-8	8270D	39	0.243	Region 5 ESL	0.081	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	310	40.4	Region 5 ESL	13	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	61	61	EPA RSL Res	20	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	12	1.6	Region 5 ESL	0.53	0.33	0.067	0.022

Matrix: Confirmatory Soil and Test Pit Soil

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
3,3'-Dichlorobenzidine	91-94-1	8270D	1.1	0.646	Region 5 ESL	0.22	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	--	3.16	Region 5 ESL	1.1	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	0.49	0.144	Region 5 ESL	0.048	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	--	--	--	--	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	610	7.95	Region 5 ESL	2.7	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	2.4	1.1	Region 5 ESL	0.37	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	31	31	EPA RSL Res	10	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	24	21.9	Region 5 ESL	7.3	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	--	5.12	Region 5 ESL	1.7	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	18	0.302	Region 5 ESL	0.1	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	0.21	0.21	EPA RSL Res	0.07	0.33	0.167	0.074
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	35	0.925	Region 5 ESL	0.31	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	260	0.239	Region 5 ESL	0.08	0.33	0.067	0.0063
Carbazole	86-74-8	8270D	--	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	7.8	7.8	EPA RSL Res	2.6	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	4900	24.8	Region 5 ESL	8.3	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	4900	734	Region 5 ESL	240	0.33	0.067	0.0065
Di-n-butyl phthalate	84-74-2	8270D	610	0.15	Region 5 ESL	0.05	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	--	709	Region 5 ESL	240	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	6.1	0.0398	Region 5 ESL	0.013	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	37	0.755	Region 5 ESL	0.25	0.33	0.067	0.032
Hexachloroethane	67-72-1	8270D	6.1	0.596	Region 5 ESL	0.2	0.33	0.167	0.096
Isophorone	78-59-1	8270D	510	139	Region 5 ESL	46	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	4.8	1.31	Region 5 ESL	0.44	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	0.069	0.069	EPA RSL Res	0.023	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	99	0.545	Region 5 ESL	0.18	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	3	2.1	EPA SSL Wildlife	0.7	0.67	0.167	0.082
Phenol	108-95-2	8270D	1800	30	ORNL Invert	10	0.33	0.067	0.029

Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Polycyclic Aromatic Hydrocarbons									
2-Methylnaphthalene	91-57-6	8270D SIM	31	29	EPA SSL Invert	9.7	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	340	20	ORNL Plant	6.7	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	340	29	EPA SSL Invert	9.7	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	1700	29	EPA SSL Invert	9.7	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.015	0.015	EPA RSL Res	0.005	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	170	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	1.5	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	15	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.015	0.015	EPA RSL Res	0.005	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	230	29	EPA SSL Invert	9.7	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	230	29	EPA SSL Invert	9.7	0.0033	0.0033	0.00057
Indeno(1,2,3-c,d)pyrene	193-39-5	8270D SIM	0.15	0.15	EPA RSL Res	0.05	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	3.6	3.6	EPA RSL Res	1.2	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	170	29	EPA SSL Invert	9.7	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	170	1.1	EPA SSL Wildlife	0.37	0.0033	0.0033	0.0017
Polychlorinated Biphenyls (PCBs)									
Aroclor-1016	12674-11-2	8082A	0.39	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.14	0.000332	Region 5 ESL	0.00011	0.033	0.0166	0.0038
Aroclor-1232	11141-16-5	8082A	0.14	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.11	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.22	0.000332	Region 5 ESL	0.00011	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	--	0.000332	Region 5 ESL	0.00011	0.033	0.0166	0.0039
Metals									
Aluminum	7429-90-5	6010C	7700	50	ORNL Plant	17	10	2.5	1.2
Antimony	7440-36-0	6010C	3.1	0.27⁽⁶⁾	EPA SSL Wildlife	0.09	1	0.35	0.18
Arsenic	7440-38-2	6020A	0.39	0.39	EPA RSL Res	0.13	0.5	0.20	0.065
Barium	7440-39-3	6010C	1500	330	EPA SSL Invert	110	10	0.7	0.38
Analyte	CAS Number	Analytical Method ⁽¹⁾	Test Pit Soil PSL = EPA RSL, Residential Soil ⁽²⁾ (mg/kg)	Confirmatory Soil PSL ⁽³⁾ (mg/kg)	Confirmatory Soil PSL Reference ⁽³⁾	Project Quantitation Limit (PQL) Goal ⁽⁴⁾ (mg/kg)	Mitkem Limits ⁽⁵⁾		
							LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)

Beryllium	7440-41-7	6010C	16	10	ORNL Plant	3.3	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	7	0.36	EPA SSL Wildlife	0.12	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	0.29	0.29 ⁽⁶⁾	EPA RSL Res	0.097	1	0.1	0.054
Cobalt	7440-48-4	6020A	2.3	2.3	EPA RSL Res	0.77	0.5	0.20	0.089
Copper	7440-50-8	6010C	310	28	EPA SSL Wildlife	9.3	1.5	0.75	0.42
Iron	7439-89-6	6010C	5500	200	ORNL Invert	67	10	5	2.2
Lead	7439-92-1	6010C	400	11	EPA SSL Wildlife	3.7	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	180	180	EPA RSL Res	60	2.5	1	0.47
Mercury	7439-97-6	7471B	2.3	0.1	ORNL Invert	0.033	0.033	0.02	0.002
Nickel	7440-02-0	6010C	150	38	EPA SSL Plant	13	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	39	0.52 ⁽⁶⁾	EPA SSL Plant	0.17	1.5	1.25	0.78
Silver	7440-22-4	6020A	39	4.2	EPA SSL Wildlife	1.4	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	--	1	ORNL Plant	0.33	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	39	2 ⁽⁶⁾	ORNL Plant	0.67	2.5	0.1	0.038
Zinc	7440-66-6	6010C	2300	46	EPA SSL Wildlife	15	2.5	0.5	0.27
Petroleum Hydrocarbons									
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	500	500	RIDEM Res TPH DEC	170	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	500	500	RIDEM Res TPH DEC	170	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.
2. The PSL for test pit soil is the EPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites, Residential Soil value, May 2010 (USEPA, 2010) except for GRO and ExTPH. The PSL for GRO and ExTPH is the RIDEM Residential TPH DEC (RIDEM, 2004). One-tenth RSLs are presented for non-carcinogens to correspond to a target hazard quotient of 0.1. Refer to Appendix H, Table H-1 for identification of non-carcinogens; references, and other notes applicable for specific analytes. Table H-1 also presents the RIDEM DEC, Residential Soil values (RIDEM 2004) for informational purposes.
3. The PSL for confirmatory soil is the lower of the EPA RSL, Residential Soil value (EPA RSL Res); or the selected ecological soil screening level (SSL). Appendix H, Table H-1 presents the EPA RSLs and all selected ecological SSLs and source references; and Appendix H, Table H-2 presents the ecological SSL source criteria and references. Refer also to Tables H-1 and H-2 for notes applicable for specific analytes
4. Although the PSLs are different for test pit soil and confirmatory soil, the LOQ Goals and the selected methods are the same for both types of samples, based on the lower of the two PSLs, in order to simplify sampling and analysis procedures.
5. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
6. Due to dilution factors required for analysis of solids by Method 6020A, a lower LOQ cannot be achieved by using Method 6020A; therefore, Method 6010C will be used.

Abbreviations:

- = Not available or not applicable

DEC = Direct Exposure Criteria

DL = Detection Limit

Eco = Ecological

EPA = Environmental Protection Agency

ESL = Ecological Screening Level

Ex = Extractable

LOD = Limit of Detection

LOQ = Limit of Quantitation

ORNL = Oak Ridge National Laboratory

Res = Residential

RIDEM = Rhode Island Department of Environmental Management

RSL = Regional Screening Level

PQL = Project Quantitation Limit

GRO = Gasoline Range Organics
 Invert = Invertebrate

TPH = Total Petroleum Hydrocarbons

SAP Worksheet #15b – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitekem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	8260C	0.0302	Region 3 BTAG	0.01	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	1.36	Region 3 BTAG	0.45	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.24	Region 3 BTAG	0.41	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	0.027	SCV	0.009	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	0.031	Region 3 BTAG	0.01	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	0.0165	Region 3 BTAG	0.0055	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.25	SCV	0.083	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	--	--	--	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	2.1	Region 3 BTAG	0.7	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	--	--	--	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	--	--	--	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	4.43	Region 3 BTAG	1.5	0.005	0.002	0.0007
1,4 Dichlorobenzene	106-46-7	8260C	0.599	Region 3 BTAG	0.2	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	0.27	SCV	0.09	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	0.022	SCV	0.0073	0.005	0.004	0.00083
4-Methyl-2-Pentanone	108-10-1	8260C	0.033	SCV	0.011	0.005	0.004	0.00073
Acetone	67-64-1	8260C	0.0087	SCV	0.0029	0.005	0.004	0.0016
Benzene	71-43-2	8260C	0.16	SCV	0.053	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	--	--	--	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	0.654	Region 3 BTAG	0.22	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	--	--	--	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	0.000851	Region 3 BTAG	0.00028	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.0642	Region 3 BTAG	0.021	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	0.00842	Region 3 BTAG	0.0028	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	--	--	--	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.022	SCV	0.0073	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	--	--	--	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	0.4	SCV	0.13	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	0.000051	SCV	0.000017	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	--	--	--	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	1.1	Region 3 BTAG	0.37	0.005	0.002	0.0005
Methylene Chloride	75-09-2	8260C	0.37	SCV	0.12	0.005	0.002	0.0013

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitekem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Styrene	100-42-5	8260C	0.559	Region 3 BTAG	0.19	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.468	Region 3 BTAG	0.16	0.005	0.002	0.00062
Toluene	108-88-3	8260C	0.05	SCV	0.017	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	1.05	Region 3 BTAG	0.35	0.005	0.002	0.00053
trans-1,3-Dichloropropene	10061-02-6	8260C	0.000051	SCV	0.000017	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	0.0969	Region 3 BTAG	0.032	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	--	--	--	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	--	--	--	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	0.16	SCV	0.053	0.005	0.002	0.00047
Semivolatile Organic Compounds								
2,4,5-Trichlorophenol	95-95-4	8270D	0.003	NOAA SQuiRT	0.001	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	0.213	Region 3 BTAG	0.071	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	0.117	Region 3 BTAG	0.039	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	0.029	Region 3 BTAG	0.0097	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	--	--	--	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	0.0416	Region 3 BTAG	0.014	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	--	--	--	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	--	--	--	0.33	0.067	0.052
2-Chlorophenol	95-57-8	8270D	0.0312	Region 3 BTAG	0.01	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	0.012	SCV	0.004	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	--	--	--	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	--	--	--	0.33	0.067	0.022
3,3'-Dichlorobenzidine	91-94-1	8270D	0.127	Region 3 BTAG	0.042	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	--	--	--	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	--	--	--	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	1.23	Region 3 BTAG	0.41	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	--	--	--	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	--	--	--	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	0.67	Region 3 BTAG	0.22	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	--	--	--	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	--	--	--	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	--	--	--	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	--	--	--	0.33	0.167	0.074
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	0.18	Region 3 BTAG	0.06	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	10.9	Region 3 BTAG	3.6	0.33	0.067	0.0063
Carbazole	86-74-8	8270D	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	0.415	Region 3 BTAG	0.14	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	0.603	Region 3 BTAG	0.2	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	0.006	NOAA SQuiRT	0.002	0.33	0.067	0.0065

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Di-n-butyl phthalate	84-74-2	8270D	6.47	Region 3 BTAG	2.2	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	0.061	NOAA SQUIRT	0.02	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	0.0013	NOAA SQUIRT	0.00043	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	--	--	--	0.33	0.067	0.032
Hexachloroethane	67-72-1	8270D	1.027	Region 3 BTAG	0.34	0.33	0.167	0.096
Isophorone	78-59-1	8270D	--	--	--	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	0.021	NOAA SQUIRT	0.007	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	--	--	--	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	2.68	Region 3 BTAG	0.89	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	0.504	Region 3 BTAG	0.17	0.67	0.167	0.082
Phenol	108-95-2	8270D	0.42	Region 3 BTAG	0.14	0.33	0.067	0.029
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	8270D SIM	0.0202	Region 3 BTAG	0.0067	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	0.0067	Region 3 BTAG	0.0022	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	0.0059	Region 3 BTAG	0.002	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	0.0572	Region 3 BTAG	0.019	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.108	Region 3 BTAG	0.036	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.15	Region 3 BTAG	0.05	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.13	NOAA SQUIRT	0.043	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	0.17	Region 3 BTAG	0.057	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	0.24	Region 3 BTAG	0.08	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	0.166	Region 3 BTAG	0.055	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.033	Region 3 BTAG	0.011	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	0.423	Region 3 BTAG	0.14	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	0.0774	Region 3 BTAG	0.026	0.0033	0.0033	0.00057
Indeno(1,2,3-c,d)pyrene	193-39-5	8270D SIM	0.017	Region 3 BTAG	0.0057	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	0.176	Region 3 BTAG	0.059	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	0.204	Region 3 BTAG	0.068	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	0.195	Region 3 BTAG	0.065	0.0033	0.0033	0.0017
Polychlorinated Biphenyls (PCBs)								
Aroclor-1016	12674-11-2	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0166	0.0038
Aroclor-1232	11141-16-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	0.0598	Region 3 BTAG	0.02	0.033	0.0166	0.0039
Metals								

Matrix: Wetland Sediment

Analyte	CAS Number	Analytical Method ⁽¹⁾	Sediment PSL (mg/kg) ⁽²⁾	Sediment PSL Reference ⁽²⁾	Project LOQ Goal (mg/kg)	Mitekem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Aluminum	7429-90-5	6010C	25500	NOAA SQuiRT	8500	10	2.5	1.2
Antimony	7440-36-0	6010C	2	Region 3 BTAG	0.67	1	0.35	0.18
Arsenic	7440-38-2	6020A	9.8	Region 3 BTAG	3.3	0.5	0.20	0.065
Barium	7440-39-3	6010C	48	NOAA SQuiRT	16	10	0.7	0.38
Beryllium	7440-41-7	6010C	--	--	--	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	0.99	Region 3 BTAG	0.33	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	43.4	Region 3 BTAG	14	1	0.1	0.054
Cobalt	7440-48-4	6020A	50	Region 3 BTAG	17	0.5	0.20	0.089
Copper	7440-50-8	6010C	31.6	Region 3 BTAG	11	1.5	0.75	0.42
Iron	7439-89-6	6010C	20000	Region 3 BTAG	6700	10	5	2.2
Lead	7439-92-1	6010C	35.8	Region 3 BTAG	12	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	460	Region 3 BTAG	150	2.5	1	0.47
Mercury	7439-97-6	7471B	0.18	Region 3 BTAG	0.06	0.033	0.02	0.002
Nickel	7440-02-0	6010C	22.7	Region 3 BTAG	7.6	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	2	Region 3 BTAG	0.67	1.5	1.25	0.78
Silver	7440-22-4	6020A	1	Region 3 BTAG	0.33	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	--	--	--	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	57	NOAA SQuiRT	19	2.5	0.1	0.038
Zinc	7440-66-6	6010C	121	Region 3 BTAG	40	2.5	0.5	0.27
Petroleum Hydrocarbons								
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	--	--	--	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	--	--	--	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.
2. The PSL is the selected ecological sediment screening level. The PSL references are presented in Appendix H, Table H-1, and the source criteria are presented in Appendix H-3.
3. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.

Acronyms and Abbreviations:

-- = Not available or not applicable
 BTAG = Biological Technical Assistance Group
 NOAA = National Oceanic and Atmospheric Administration
 SCV = Secondary Chronic Value
 Sed = Sediment
 SQB = Sediment Quality Benchmarks
 SQUIRT = Screening Quick Reference Tables
 USEPA = United States Environmental Protection Agency

SAP Worksheet #15 – Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	8260C	0.0302	Sed PSL	0.01	0.005	0.002	0.00053
1,1,2,2-Tetrachloroethane	79-34-5	8260C	0.127	CS PSL	0.042	0.005	0.002	0.00068
1,1,2-Trichloroethane	79-00-5	8260C	1.1	CS PSL	0.37	0.005	0.002	0.00048
1,1-Dichloroethane	75-34-3	8260C	0.027	Sed PSL	0.009	0.005	0.002	0.00067
1,1-Dichloroethene	75-35-4	8260C	0.031	Sed PSL	0.01	0.005	0.002	0.00095
1,2-Dichlorobenzene	95-50-1	8260C	0.0165	Sed PSL	0.0055	0.005	0.002	0.00062
1,2-Dichloroethane	107-06-2	8260C	0.25	Sed PSL	0.083	0.005	0.002	0.00054
1,2-Dichloropropane	78-87-5	8260C	0.89	CS PSL	0.3	0.005	0.002	0.00069
1,2,4-Trichlorobenzene	120-82-1	8260C	2.1	Sed PSL	0.7	0.005	0.002	0.00063
1,2,4-Trimethylbenzene	95-63-6	8260C	6.2	CS PSL	2.1	0.005	0.002	0.00057
1,3,5-Trimethylbenzene	108-67-8	8260C	78	CS PSL	26	0.005	0.002	0.00061
1,3-Dichlorobenzene	541-73-1	8260C	4.43	Sed PSL	1.5	0.005	0.002	0.0007
1,4-Dichlorobenzene	106-46-7	8260C	0.546	CS PSL	0.18	0.005	0.002	0.0008
2-Butanone	78-93-3	8260C	0.27	Sed PSL	0.09	0.005	0.004	0.002
2-Hexanone	591-78-6	8260C	0.022	Sed PSL	0.0073	0.005	0.004	0.00083
4-Methyl-2-Pentanone	108-10-1	8260C	0.033	Sed PSL	0.011	0.005	0.004	0.00073
Acetone	67-64-1	8260C	0.0087	Sed PSL	0.0029	0.005	0.004	0.0016
Benzene	71-43-2	8260C	0.16	Sed PSL	0.053	0.005	0.002	0.00061
Bromodichloromethane	75-27-4	8260C	0.27	CS PSL	0.09	0.005	0.002	0.00097
Bromoform	75-25-2	8260C	0.654	Sed PSL	0.22	0.005	0.002	0.002
Bromomethane	74-83-9	8260C	0.235	CS PSL	0.078	0.005	0.002	0.0011
Carbon Disulfide	75-15-0	8260C	0.000851	Sed PSL	0.00028	0.005	0.002	0.0003
Carbon Tetrachloride	56-23-5	8260C	0.0642	Sed PSL	0.021	0.005	0.002	0.00033
Chlorobenzene	108-90-7	8260C	0.00842	Sed PSL	0.0028	0.005	0.002	0.00051
Chloroethane	75-00-3	8260C	1500	CS PSL	500	0.005	0.002	0.001
Chloroform	67-66-3	8260C	0.022	Sed PSL	0.0073	0.005	0.002	0.00064
Chloromethane	74-87-3	8260C	10.4	CS PSL	3.5	0.005	0.002	0.0008
cis-1,2-Dichloroethene	156-59-2	8260C	0.4	Sed PSL	0.13	0.005	0.002	0.00075
cis-1,3-Dichloropropene	10061-01-5	8260C	0.000051	Sed PSL	0.000017	0.005	0.002	0.00067
Dibromochloromethane	124-48-1	8260C	0.68	CS PSL	0.23	0.005	0.002	0.00065
Ethylbenzene	100-41-4	8260C	1.1	Sed PSL	0.37	0.005	0.002	0.0005
Methylene Chloride	75-09-2	8260C	0.37	Sed PSL	0.12	0.005	0.002	0.0013
Styrene	100-42-5	8260C	0.559	Sed PSL	0.19	0.005	0.002	0.00052
Tetrachloroethene	127-18-4	8260C	0.468	Sed PSL	0.16	0.005	0.002	0.00062
Toluene	108-88-3	8260C	0.05	Sed PSL	0.017	0.005	0.002	0.00047
trans-1,2-Dichloroethene	156-60-5	8260C	0.784	CS PSL	0.26	0.005	0.002	0.00053

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
trans-1,3-Dichloropropene	10061-02-6	8260C	0.000051	Sed PSL	0.000017	0.005	0.002	0.00068
Trichloroethene	79-01-6	8260C	0.0969	Sed PSL	0.032	0.005	0.002	0.00062
Trichlorofluoromethane (CFC-11)	75-69-4	8260C	16.4	CS PSL	5.5	0.005	0.004	0.00042
Vinyl Chloride	75-01-4	8260C	0.06	CS PSL	0.02	0.005	0.002	0.00063
Xylenes (total)	1330-20-7	8260C	0.16	Sed PSL	0.053	0.005	0.002	0.00047
Semivolatile Organic Compounds								
2,4,5-Trichlorophenol	95-95-4	8270D	0.003	Sed PSL	0.001	0.67	0.067	0.053
2,4,6-Trichlorophenol	88-06-2	8270D	0.213	Sed PSL	0.071	0.33	0.067	0.026
2,4-Dichlorophenol	120-83-2	8270D	0.117	Sed PSL	0.039	0.33	0.067	0.017
2,4-Dimethylphenol	105-67-9	8270D	0.01	CS PSL	0.0033	0.33	0.067	0.053
2,4-Dinitrophenol	51-28-5	8270D	0.0609	CS PSL	0.02	0.67	0.167	0.1
2,4-Dinitrotoluene	121-14-2	8270D	0.0416	Sed PSL	0.014	0.33	0.067	0.006
2,6-Dinitrotoluene	606-20-2	8270D	0.0328	CS PSL	0.011	0.33	0.067	0.0088
2-Chloronaphthalene	91-58-7	8270D	0.0122	CS PSL	0.0041	0.33	0.067	0.052
2-Chlorophenol	95-57-8	8270D	0.0312	Sed PSL	0.01	0.33	0.067	0.017
2-Methylphenol	95-48-7	8270D	0.012	Sed PSL	0.004	0.33	0.067	0.023
2-Nitroaniline	88-74-4	8270D	61	CS PSL	20	0.67	0.067	0.0055
2-Nitrophenol	88-75-5	8270D	1.6	CS PSL	0.53	0.33	0.067	0.022
3,3'-Dichlorobenzidine	91-94-1	8270D	0.127	Sed PSL	0.042	0.33	0.067	0.052
3-Nitroaniline	99-09-2	8270D	3.16	CS PSL	1.1	0.67	0.067	0.022
4,6-Dinitro-2-methylphenol	534-52-1	8270D	0.144	CS PSL	0.048	0.67	0.067	0.03
4-Bromophenyl phenyl ether	101-55-3	8270D	1.23	Sed PSL	0.41	0.33	0.067	0.067
4-Chloro-3-methylphenol	59-50-7	8270D	7.95	CS PSL	2.7	0.33	0.067	0.02
4-Chloroaniline	106-47-8	8270D	1.1	CS PSL	0.37	0.33	0.067	0.017
4-Chlorophenyl phenyl ether	7005-72-3	8270D	--	--	--	0.33	0.067	0.015
4-Methylphenol	106-44-5	8270D	0.67	Sed PSL	0.22	0.33	0.067	0.022
4-Nitroaniline	100-01-6	8270D	21.9	CS PSL	7.3	0.67	0.067	0.025
4-Nitrophenol	100-02-7	8270D	5.12	CS PSL	1.7	0.67	0.067	0.018
Bis(2-Chloroethoxy)methane	111-91-1	8270D	0.302	CS PSL	0.1	0.33	0.067	0.021
bis(2-Chloroethyl)ether	111-44-4	8270D	0.21	CS PSL	0.07	0.33	0.167	0.074
Bis(2-Ethylhexyl)phthalate	117-81-7	8270D	0.18	Sed PSL	0.06	0.33	0.067	0.0086
Butyl benzyl phthalate	85-68-7	8270D	0.239	CS PSL	0.08	0.33	0.067	0.0063
Carbazole	86-74-8	8270D	--	--	--	0.33	0.067	0.01
Dibenzofuran	132-64-9	8270D	0.415	Sed PSL	0.14	0.33	0.067	0.0058
Diethyl phthalate	84-66-2	8270D	0.603	Sed PSL	0.2	0.33	0.067	0.008
Dimethyl phthalate	131-11-3	8270D	0.006	Sed PSL	0.002	0.33	0.067	0.0065
Di-n-butyl phthalate	84-74-2	8270D	0.15	CS PSL	0.05	0.33	0.067	0.0058
Di-n-octyl phthalate	117-84-0	8270D	0.061	Sed PSL	0.02	0.33	0.067	0.044
Hexachlorobutadiene	87-68-3	8270D	0.0013	Sed PSL	0.00043	0.33	0.067	0.032
Hexachlorocyclopentadiene	77-47-4	8270D	0.755	CS PSL	0.25	0.33	0.067	0.032

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Hexachloroethane	67-72-1	8270D	0.596	CS PSL	0.2	0.33	0.167	0.096
Isophorone	78-59-1	8270D	139	CS PSL	46	0.33	0.067	0.051
Nitrobenzene	98-95-3	8270D	0.021	Sed PSL	0.007	0.33	0.067	0.018
N-Nitrosodi-n-propylamine	621-64-7	8270D	0.069	CS PSL	0.023	0.33	0.167	0.068
N-Nitrosodiphenylamine	86-30-6	8270D	0.545	CS PSL	0.18	0.33	0.067	0.047
Pentachlorophenol	87-86-5	8270D	0.504	Sed PSL	0.17	0.67	0.167	0.082
Phenol	108-95-2	8270D	0.42	Sed PSL	0.14	0.33	0.067	0.029
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	8270D SIM	0.0202	Sed PSL	0.0067	0.0033	0.00083	0.00025
Acenaphthene	83-32-9	8270D SIM	0.0067	Sed PSL	0.0022	0.0033	0.00083	0.00029
Acenaphthylene	208-96-8	8270D SIM	0.0059	Sed PSL	0.002	0.0033	0.00083	0.00024
Anthracene	120-12-7	8270D SIM	0.0572	Sed PSL	0.019	0.0033	0.0033	0.0025
Benzo(a)anthracene	56-55-3	8270D SIM	0.108	Sed PSL	0.036	0.0033	0.0033	0.0012
Benzo(a)pyrene	50-32-8	8270D SIM	0.015	CS PSL	0.005	0.0033	0.0033	0.00077
Benzo(b)fluoranthene	205-99-2	8270D SIM	0.13	Sed PSL	0.043	0.0033	0.0033	0.0018
Benzo(g,h,i)perylene	191-24-2	8270D SIM	0.17	Sed PSL	0.057	0.0033	0.0033	0.00086
Benzo(k)fluoranthene	207-08-9	8270D SIM	0.24	Sed PSL	0.08	0.0033	0.0033	0.0011
Chrysene	218-01-9	8270D SIM	0.166	Sed PSL	0.055	0.0033	0.0033	0.00094
Dibenzo(a,h)anthracene	53-70-3	8270D SIM	0.015	CS PSL	0.005	0.0033	0.0033	0.00069
Fluoranthene	206-44-0	8270D SIM	0.423	Sed PSL	0.14	0.0033	0.0033	0.002
Fluorene	86-73-7	8270D SIM	0.0774	Sed PSL	0.026	0.0033	0.0033	0.00057
Indeno(1,2,3-cd)pyrene	193-39-5	8270D SIM	0.017	Sed PSL	0.0057	0.0033	0.0033	0.00072
Naphthalene	91-20-3	8270D SIM	0.176	Sed PSL	0.059	0.0033	0.00083	0.0007
Phenanthrene	85-01-8	8270D SIM	0.204	Sed PSL	0.068	0.0033	0.0033	0.0026
Pyrene	129-00-0	8270D SIM	0.195	Sed PSL	0.065	0.0033	0.0033	0.0017
Polychlorinated Biphenyls (PCBs)								
Aroclor-1016	12674-11-2	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0023
Aroclor-1221	11104-28-2	8082A	0.000332	CS PSL	0.00011	0.033	0.0166	0.0038
Aroclor-1232	11141-16-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0011
Aroclor-1242	53469-21-9	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0023
Aroclor-1248	12672-29-6	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0029
Aroclor-1254	11097-69-1	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0039
Aroclor-1260	11096-82-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0016
Aroclor-1262	37324-23-5	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0015
Aroclor-1268	11100-14-4	8082A	0.000332	CS PSL	0.00011	0.033	0.0083	0.0015
PCBs (Total)	1336-36-3	8082A	0.000332	CS PSL	0.00011	0.033	0.0166	0.0039
TAL Metals								
Aluminum	7429-90-5	6010C	50	CS PSL	17	10	2.5	1.2
Antimony	7440-36-0	6010C	0.27⁽⁴⁾	CS PSL	0.09	1	0.35	0.18
Arsenic	7440-38-2	6020A	0.39	CS PSL	0.13	0.5	0.20	0.065

Matrix: Residual Material

Analyte	CAS Number	Analytical Method ⁽¹⁾	Lower of Confirmatory Soil or Sediment PSL ⁽²⁾ (mg/kg)	Reference of Lower of Confirmatory Soil or Sediment PSL ⁽²⁾	Project LOQ Goal (mg/kg)	Mitkem Limits ⁽³⁾		
						LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Barium	7440-39-3	6010C	48	Sed PSL	16	10	0.7	0.38
Beryllium	7440-41-7	6010C	10	CS PSL	3.3	0.25	0.005	0.002
Cadmium	7440-43-9	6010C	0.36	CS PSL	0.12	0.25	0.025	0.013
Calcium	7440-70-2	6010C	--	--	--	40	12.5	6.6
Chromium	7440-47-3	6010C	0.29⁽⁴⁾	CS PSL	0.097	1	0.1	0.054
Cobalt	7440-48-4	6020A	2.3	CS PSL	0.77	0.5	0.20	0.089
Copper	7440-50-8	6010C	28	CS PSL	9.3	1.5	0.75	0.42
Iron	7439-89-6	6010C	200	CS PSL	67	10	5	2.2
Lead	7439-92-1	6010C	11	CS PSL	3.7	0.5	0.25	0.14
Magnesium	7439-95-4	6010C	--	--	--	25	3	1.5
Manganese	7439-96-5	6010C	180	CS PSL	60	2.5	1	0.47
Mercury	7439-97-6	7471B	0.1	CS PSL	0.033	0.033	0.02	0.002
Nickel	7440-02-0	6010C	22.7	Sed PSL	7.6	2.5	0.125	0.066
Potassium	7440-09-7	6010C	--	--	--	50	5	2.4
Selenium	7782-49-2	6010C	0.52⁽⁴⁾	CS PSL	0.17	1.5	1.25	0.78
Silver	7440-22-4	6020A	1	Sed PSL	0.33	0.5	0.20	0.038
Sodium	7440-23-5	6010C	--	--	--	50	2	0.91
Thallium	7440-28-0	6020A	1	CS PSL	0.33	0.5	0.20	0.064
Vanadium	7440-62-2	6010C	2⁽⁴⁾	CS PSL	0.67	2.5	0.1	0.038
Zinc	7440-66-6	6010C	46	CS PSL	15	2.5	0.5	0.27
Petroleum Hydrocarbons								
Gasoline Range Organics (C ₅ -C ₁₂)	-	8015D	500	CS PSL	170	2.5	1	0.45
Extractable TPH (C ₉ -C ₄₀)	-	8015D	500	CS PSL	170	12	1.7	1.3

Notes:

1. All methods are EPA SW-846.
2. PSLs are not applicable for residual material (see Section 11.2) because residual material concentrations will be compared with downgradient soil and sediment concentrations rather than with screening material. However, in order to determine the analytical methods and laboratory LOQs and LODs needed to allow comparison of residual material concentrations with detected levels of soil and sediment, values equal to the lower of the PSLs for confirmatory soil and sediment are presented. Those PSLs and their references are presented in Appendix H, Table H-1.
3. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
4. Due to dilution factors required for analysis of solids by Method 6020A, a lower LOQ cannot be achieved by using Method 6020A; therefore, Method 6010C will be used.

Abbreviations:

-- = Not available or not applicable
 CS = Confirmatory Soil
 Sed = Sediment

ATTACHMENT B
DATA PACKAGE DELIVERABLES REQUIREMENTS

DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide a hard copy plus two compact disks (CDs) each containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP-like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, pesticide, PCBs, TPH, metals, pH, and TOC, etc., as applicable)
 - a. Results and QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
 - b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
 - c. Calibration Data (includes all initial and continuing calibrations and second-source initial calibration verifications)
 - d. Miscellaneous (includes extraction/preparation forms, percent solids determination, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

In addition to the following forms, second-source initial calibration verification summary forms are required, if applicable per the DOD QSM.

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

Results Report - must present all information presented on CLP FORM 1 (ILM05.4).

Initial and Continuing Calibration Summary - must present all information presented on CLP FORM 2A (ILM05.4).

Low-Level Calibration Standard Summary – if applicable, must present all information presented on CLP FORM 2B (ILM05.4).

Blanks - must present all information presented on CLP FORM 3 (ILM05.4).

ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4 (ILM05.4).

Matrix Spike Summary - must present all information presented on CLP FORM 5A (ILM05.4).

Post Digestion Spike - must present all information presented on CLP FORM 5B (ILM05.4).

Lab Duplicate Results - must present all information presented on CLP FORM 6 (ILM05.4).

LCS Summary - must present all information presented on CLP FORM 7 (ILM05.4).

MSA Summary (Method of Standard Addition) – if applicable, must present all information presented on CLP FORM 8 (ILM04.1).

ICP Serial Dilution Summary - must present all information presented on CLP FORM 8 (ILM05.4).

Detection Limits - must present all information presented on CLP FORM 9 (ILM05.4).

Linear Range – must present all information presented on CLP FORM 11 (ILM05.4).

Internal Standard Association (ICP-MS) – must present all information presented on CLP FORM 11 (ISM01.2). Alternatively, the laboratory may provide the information in the Narrative.

Prep Log - must present all information presented on CLP FORM 12 (ILM05.4).

Analysis Run Log - must present all information presented on CLP FORM 13 (ILM05.4).

ICP-MS Tune – must present all information presented on CLP FORM 14 (ISM01.2). Laboratory must also document the number of tune analysis integrations on this form or in the Narrative.

ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

In addition, the Narrative, summary forms, or raw data must indicate the number of replicate integrations for ICP-MS sample analysis.

Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

Summary Forms for SW-846 8081A and 8082 Pesticide and Polychlorinated Biphenyl (PCB) Organic Compounds (and 8151A, 8141A, and all other SW-846 GC methods) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Pesticide Initial Calibration of Single Component Analytes	Present all information contained on CLP Form VI-PEST-2.
Summary of Pesticide Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Pesticide Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Pesticide Identification Summary For Single Component Analytes and for Multiple Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: <http://www.lab-data.com>. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at _____ if there are any questions or problems with the enclosed electronic deliverables.

Signature: _____ Title: _____ Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field “ClientSampleID” will always be the first piece of information to start every new line of data (or database record), followed by the field “LabAnalysisRefMethodID”, “AnalysisType”, etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the “Collected”, “ShippingBatchID”, and “Temperature” fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields (“ProjectNumber” and “ProjectName”) are omitted in this example.

...“LCSW100598” “AQ” “LCSW100598” “LCS” “8270C”,...etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the “MatrixID” field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetrtech.com

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor’s identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID	Text	25	X

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field.</p> <p>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01<u>DL</u> and MW01<u>RE</u> are not allowed.</p> <p>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.</p>			
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and	Text	25	X

Table A1**Field Descriptions for the Analytical Results Table (Table A1)**

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>that the LabSampleID must be distinct for a given field sample or lab QC sample and method.</p> <p>Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.</p>			
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	<p>CAS Number or unique client identifier for an analyte or isotope.</p> <p>If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes.</p> <p>For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.)</p> <p>For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.</p>	Text	12	X
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as	Text	60	X

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	TIC's).			
Result	<p>Result value for the analyte or isotope.</p> <p>Entries must be numeric. For non-detects of target analytes or isotopes and spikes, do not enter "ND" or "0". Do not leave this field blank. If an analyte or spike was not detected, enter the associated value specified in Section 3.0 of this scope of work (e.g., LOD, SQL, PQL, etc.), corrected for dilution and percent moisture as applicable. Do not enter "0". A "0" result may be acceptable for surrogate or internal standard percent recoveries; however, it should not be reported for any target compound.</p>	Numeric ⁽¹⁾	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	<p>A string of single letter result qualifiers assigned by the lab based on client-defined rules and values.</p> <p><u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.</p>	Text	7	Q

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
DetectionLimit	<p>For radiochemistry methods, the minimum detectable activity for the isotope being measured.</p> <p>For all other methods: The minimum detection limit value for the analyte being measured.</p> <p>For surrogates, internal standards, etc. where detection limits are not applicable use the value -99.</p>	Numeric ⁽¹⁾	10(6)	X
DetectionLimitType	<p>Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).</p> <p>If -99 is specified in the DetectionLimit field us the value NA.</p>	Text	10	X
RetentionTime or Error	<p><u>For radiochemistry methods only</u>, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field.</p> <p><u>For GC/MS methods only</u>, enter the time expressed in decimal minutes between injection and detection for <u>GC/MS TICs only</u></p> <p><u>For target analytes in all other methods</u>, leave this field blank. Note: GC retention times are not evaluated at this time.</p>	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	<p>For radiochemistry methods: The tracer yield, if applicable.</p> <p>For all other analytical methods: The percent recovery value of a spiked</p>	Numeric ⁽¹⁾	5(3)	X

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>compound or surrogate.</p> <p>If the spike or surrogate was not recovered because of dilution, enter "DIL". If a spike or surrogate was not recovered because of matrix interference, enter "INT". If a spike or surrogate was not recovered because it was not added to the sample, enter "NS".</p>			
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric ⁽¹⁾	5(3)	X
ReportingLimit	<p>Reporting limit value for the measured analyte or isotope</p> <p>Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP.</p> <p>For surrogates, internal standards, etc. where reporting limits are not applicable use the value -99.</p>	Numeric ⁽¹⁾	10(6)	X
ReportingLimitType	<p>Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.</p> <p>If -99 is specified in the ReportingLimit field us the value NA.</p>	Text	10	X
ReportableResult	This field indicates whether or not the	Text	3	X

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>laboratory chooses an individual analyte or isotope result as reportable. Enter “YES” if the result is reportable. Enter “NO” if the result is not reportable.</p> <p>If only one analysis is submitted for a particular sample and method, enter “YES” for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC).</p> <p>If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter “YES” from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter “NO” for benzene and enter “YES” for all other compounds. For the diluted analysis (Analysis Type = DL), enter “YES” for benzene and enter “NO” for all other compounds.</p> <p>For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter “YES” for all TICs. In the other analysis enter “NO” for all TICs.</p>			

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.</p>			
SpkConcnAdded	<p>The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: $\% \text{ Recovery} = (\text{SSA} - \text{SC}) / \text{SA} \times 100\%$ where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount. Enter -99 where no spike was added.</p>	Numeric ⁽¹⁾	10(6)	X
SpkParentSampleID	<p>The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking.</p> <p>The result for SpkParentSampleID and the</p>	Text	25	X

Table A1

Field Descriptions for the Analytical Results Table (Table A1)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.			
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric ⁽¹⁾	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric ⁽¹⁾	20(6)	
LimitOfDetection	The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value \geq DL) and a 1% chance of reporting a non-detect (a false negative).	Numeric ⁽¹⁾	10(6)	N
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.

Q Only required if laboratory has qualified the result.

T Only required for tentatively identified compounds by GC/MS.

(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	<p>Client or contractor's identifier for a field sample</p> <p>If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Laboratory QC samples (i.e. Method Blanks, LCS, and LCSD, etc.) enter the unique LaboratorySampleID into this field</p> <p>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01<u>DL</u> and MW01<u>RE</u> are not allowed</p> <p>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.</p>	Text	25	X
Collected	<p>Date and Time of sample collection. Refer to the date/time format at the end of this table.</p> <p>Leave this field blank for Method Blank, LCS, and LCSD. For Collected values that are not applicable use the value of 00/00/0000 00:00.</p>	Date/Time	16*	X
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
LabSampleID	<p>Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD.</p> <p>There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.</p>	Text	25	X
QCType	<p>This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). <u>For regular environmental samples, populate this field with "NM".</u></p>	Text	10	X
ShippingBatchID	<p>Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.</p>	Text	25	X
Temperature	<p>Temperature (in centigrade degrees) of the sample as received.</p> <p>The storage refrigerator or room temperature should be reported (in centigrade degrees) for laboratory QC samples (i.e. method blanks, laboratory control standards).</p> <p>Use -99 if temperature is not available.</p> <p><u>This field is not required for radiochemistry methods.</u></p>	Numeric ⁽¹⁾	10(6)	X
LabAnalysisRefMethodID	<p>Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a</p>	Text	25	X

Table A3**Field Description for the Sample Analysis (Table A3)**

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.			
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use "Gen Prep".	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	Refer to the date/time format at the end of this table. If no sample preparation is involved enter the analysis date and time in this field. Refer to the date/time format at the end of this table.	Date/Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table. For Analyzed values that are not applicable use the value of 00/00/0000 00:00.	Date/Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted	Text	6	X

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	for the sample, enter "COA". If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter "COCAL"			
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. Enter "WET" if results are not corrected for percent moisture. Enter "DRY" if percent moisture correction is applied to results. For aqueous samples, enter "WET". For other matrices where basis is not applicable enter "NA"	Text	3	X
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. If not applicable please report "NA"	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric ⁽¹⁾	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Enter "NA" if the sample analysis was <u>not</u> performed on a leachate.	Text	10	X
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Enter "NA" if the sample analysis was <u>not</u> performed on a leachate.	Text	12	X

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. . For Analyzed values that are not applicable use the value of 00/00/0000 00:00	Date /Time	16*	X
Percent_Moisture	For soil and sediment samples, enter the percent of sample composed of water. For aqueous samples enter "100". For other matrices where Percent_Moisture is not applicable use a value of -99	Numeric ⁽¹⁾	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated	Text	12	X

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	samples.			
RunBatch	<p><u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method</p> <p>The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2.</p> <p>If Table A2 is not submitted enter a value of 'NA" in this field.</p>	Text	12	X
AnalysisBatch	<p><u>For radiochemistry methods</u> leave this field blank.</p> <p><u>For all other methods</u> the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the</p>	Text	12	X

Table A3

Field Description for the Sample Analysis (Table A3)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	<p>AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method</p> <p>The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.</p>			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.

X Required field.

(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access. To access the site or begin the registration process, visit the ISG web site at <http://isg.ttnus.com> and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using their credentials. New users must select the "Register" button and provide all of the requested information.

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the “Upload/Check Files” link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetrattech.com, and provide a full description of your organization name, contact information and include “Laboratory Contractor ID Request” in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the “Browse” button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the “Upload” button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the “Check Files” button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the “Check Files” button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the "Submit Files" button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

APPENDIX F
PROJECT-SPECIFIC FIELD TASK PROCEDURES

APPENDIX F

PROJECT-SPECIFIC FIELD TASK PROCEDURES FOR SOIL SAMPLE AND RESIDUAL MATERIAL SAMPLE COLLECTION IR SITE 03/QDC OUTFALL 001 FORMER NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Project-specific sample collection procedures for the confirmatory samples, test pit samples, and residual material samples to be collected at QDC Outfall 001 are presented below. All samples will be sent for laboratory analysis of VOCs, SVOCs, GRO, ExTPH, PAHs, PCBs, and TAL metals. Separate procedures are presented below for Volatile Analytical Parameters (VOCs/GRO) and for Non-Volatile Analytical Parameters (SVOCs, ExTPH, PAHs, PCBs, and TAL metals), as well as for collection of a sample for percent moisture. The field document forms are included in Appendix C and the sampling SOPs referenced below are included in Appendix D.

F.1 Confirmatory Soil Samples Collected From Excavation Area

Five 5-point composite soil samples will be collected from the excavation area downstream from the drain pipe outlet. Two excavation bottom samples and three sidewall samples will be collected from the locations shown on Figure 17-1. The soil sample aliquots will be collected accordance with SOP SA-1.3 using hand augers, decontaminated stainless steel trowels, disposable plastic scoops, or disposable pre-cut syringes.

For *sidewall samples*, surficial soil will be scraped away and sample collected from the area immediately beneath. Excavation *bottom samples* will be collected from a depth of 6 to 12 inches bgs to ensure that native soils at the bottom of the excavation area are evaluated rather than soils that have accumulated in the excavation due to erosion or precipitation runoff entering the excavation.

Samples for VOC and GRO analysis will be collected directly from the sidewall/bottom of the excavation and extruded into pre-preserved VOC vials. Samples for the volatile analytical parameters will not be subject to homogenization as described in the following paragraph. An aliquot will be collected for field screening analysis using the photoionization detector (PID) jar headspace technique using similar techniques. See Section F.6 for field screening procedures. Soils that are field screened using the jar headspace technique will be managed as investigation derived waste (IDW) and not containerized for laboratory analysis.

For the non-volatile analyses, the five soil aliquots that will make up each composite sample will be combined in a stainless steel bowl and thoroughly homogenized to create one composite sample. After

the composite sample is completely homogenized, soils will be transferred to the appropriate sample containers for the non-volatile laboratory analyses.

All sample collection devices will be discarded (if disposable) after collection of each sample or decontaminated between sampling locations in order to prevent cross-contamination between sampling locations. Decontamination procedures are described in Section F.7.

Refer to Section F.5 for volatile and non-volatile soil sample collection procedures.

F.2 Sediment Sample Collection From Downgradient Wetland

Sediment samples will be collected from five locations within the wetland area located downgradient from the outfall excavation area as shown on Figure 17-1. It is anticipated that sediment samples will be collected in dry conditions. Therefore, sediment samples will be collected from 0- to 6-inches below ground surface using decontaminated stainless steel trowels, disposable plastic scoops, or disposable pre-cut syringes.

Sample aliquots for volatile analytical parameters will be collected before non-volatile analyses using a disposable pre-cut syringe as described in Section F.5 below. Assuming the moisture content of the sediments is low enough, a sediment sample will be collected for PID jar headspace field screening using similar techniques. Sediments that are field screened using the jar headspace technique will be managed as IDW and not containerized for laboratory analysis.

After the collection of soil aliquots for volatile analytical parameters and jar headspace field screening, soil aliquots will be collected for non-volatile analytical parameters.

All sample collection devices will be discarded (if disposable) after collection of each sample or decontaminated between sampling locations in order to prevent cross-contamination between sampling locations. Decontamination procedures are described in Section F.7.

Refer to Section F.5 for volatile and non-volatile soil sample collection procedures.

F.3 Subsurface Soil Sample Collection From Test Pits

Four test pits will be excavated at selected locations adjacent to the drain line as described in Section 17.1.2 of the SAP and depicted on Figure 17-2. The locations will be selected after completion of the

drainage pipe reconnaissance. One soil sample will be collected from each test pit for laboratory analysis.

Test pits will be excavated in accordance with the procedures described in SOP SA-1.3 using a rubber tired backhoe or equivalent machine. The anticipated depth of excavation is between 3 and 5 feet, therefore test pits will not be accessed by sampling personnel. Soil sample collection will be accomplished using the excavator bucket or remote sampler consisting of a steel conduit with a sample collection device attached. All non-disposable sampling equipment (including the excavator bucket) will be decontaminated prior to collecting samples and in between each sample location to prevent cross contamination between sampling locations as described in Section F.7.

Soil samples will be collected from the material located adjacent to and below the pipe, to the extent practical, in order to evaluate whether contaminants have been released from the pipe to the subsurface soils. One soil sample will be collected from each test pit for laboratory analysis. Grab samples will be collected for field screening and VOC/GRO laboratory analysis according to the sampling procedures below for volatile analytical parameters (Section F.5.1). The VOC/GRO sample will be collected from the most heavily contaminated portion of the sample, based on the field screening results and/or visual observations. Soils that are field screened using the jar headspace technique will be managed as IDW and not containerized for laboratory analysis. The specific depth of the soil sample will be recorded on a test pit log sheet. Soil samples for non-volatile analytical parameters will be collected after the collection of field screening and volatile analytical parameter samples, as described in Section F.5.3.

After soil samples are collected, test pits will be backfilled with native material to the original grade and compacted using the excavator bucket.

F.4 Residual Material Samples Collected from Within Drain Pipe

Residual material samples will be collected from within the pipeline, to the extent practical, at each test pit location where an area of compromised pipe is encountered and residual material is able to be recovered. Residual material samples will be collected in the same manner as described above for soil samples collected from test pits.

One residual material sample will be collected from within the first 2 feet of the interior of the drain pipe. The sample will be collected using a decontaminated stainless steel trowel, disposable plastic scoop, or pre-cut syringe. Sample aliquots for field screening and volatile analytical parameters will be collected before non-volatile analytical parameters as described above for soil samples collected from the excavation area. After the collection of samples for field screening and volatile analytical parameters,

sufficient sample volume for all of the non-volatile analytical parameters will be collected and transferred to a stainless steel bowl and thoroughly homogenized.

Sample aliquots will be collected for laboratory analysis following the volatile and non-volatile procedures summarized in Section F.5.

F.5 Detailed Soil Sample Collection Procedures

This section describes sample collection procedures for volatile and non-volatile constituents.

F.5.1 Soil Sampling Procedures for Volatile Analytical Parameters (VOC/GRO)

Each soil sample for VOC/GRO analysis will be collected using a cut syringe or equivalent device. VOC samples will consist of two vials containing reagent-grade water, frozen within 48 hours of sample collection. GRO samples will consist of one methanol-preserved vial according to the SW-846 Method 5035A (July 2002). The VOC samples will be frozen for up to 14 days until analysis, and the methanol-preserved vials will be maintained at 4°C for up to 14 days until analysis. The following procedures will be followed for the soil VOC/GRO sample collection:

1. Label the following sample containers with the sample location number and a bottle letter such as A, B, etc.: two 40-mL amber vials containing 5 mL of reagent-grade water for VOC samples and one 40-mL amber vial containing 5 mL methanol for the GRO aliquot.
2. Collect approximately 5 grams of sample by coring or stabbing the soil with a 10-mL pre-cut syringe. Extrude the sample into one of the 40-mL VOC vials containing 5 mL of water or methanol. The soil must be immersed in the water/methanol; recollect the sample using a smaller volume if necessary. Avoid touching the threads on the vial's neck or loss of water or methanol by evaporation. Cap the vial and invert it several times to mix the sample.
3. If samples are to be shipped via next day air, weigh each sample vial to the nearest 0.01 gram and record the weight on the VOC soil sample collection/preservation log sheet. Repeat the sample collection procedure for the remaining vials. Pack and ship to the laboratory. Include the field log sheet containing the sample weight information with the samples. If samples are delivered to the analytical laboratory by ground courier, the weight of the VOC vial will not be recorded after sample collection.

Field duplicate samples will also be collected. Following the collection of the first set of VOC/GRO containers, collect the field duplicate from the same sampling interval.

For laboratory QC analyses, collect triple volume of soil samples for VOC/GRO analyses by filling three times as many sample containers.

F.5.2 Soil Sample for Percent Moisture

Fill one 2-oz. container with soil representing the same locations where the 40-mL VOC/GRO sample vials were collected. Every effort should be made to obtain the percent moisture soil aliquot as close as possible to the location where the VOC/GRO samples were collected.

F.5.3 Soil Sampling Procedures for Non-Volatile Parameters

Soil samples for non-volatile parameters will be collected using the following procedures:

1. Record all required data on the soil sample log sheet (Appendix C). Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. The soil sample log sheet will also contain soil descriptions, sample depth intervals, and other pertinent observations relative to the potential presence of contamination. The soil will be visually classified using the Unified Soil Classification System (ASTM D-2488-98).
2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.
3. Collect soil aliquots into a decontaminated stainless-steel bowl using only disposable plastic scoops or decontaminated stainless steel trowels, and homogenize the sample.
4. If there is insufficient sample volume to fill all the containers for the non-volatile analyses, then collect additional soil aliquots adjacent to the initial locations to obtain the minimum sample volume required for laboratory analysis.
5. Remove any large particles such as gravel or artificial fill too large to be sent for analysis (typically greater than a common green pea in dimensions). Note the removal of material on the boring log.
6. Fill the appropriate sample containers.

7. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.
8. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the chain-of-custody procedures described in Worksheet 27 are followed. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.
9. Decontaminate the sampling equipment before reuse (SOP SA-7.1).

Care should be taken in handling all soil samples to ensure that the exterior of the sample containers are clean and free of soils before shipping to the laboratory.

All laboratory analytical samples will be kept on ice in coolers and will be shipped with appropriate QA/QC samples.

F.6 PID Jar Headspace Technique

Field screening of soil samples will be conducted using the following procedure:

1. Soil or "mason" type jars with a volume of 16 oz. (approximately 500 ml) are preferred. Jars with a volume of less than 8 oz. (approximately 250 ml) should not be used.
2. Half-fill a clean glass jar with the sample to be analyzed. Quickly cover the open top with one or two sheets of clean aluminum foil. Replace screw cap and tightly seal the jar.
3. Vigorously shake jars for 15 seconds. Allow headspace to develop for at least 5 minutes. When temperatures are below 32° F, headspace development should be done in a heated vehicle or building. Vigorously shake jars again for 15 seconds.
4. Remove screw-lid and expose the foil. Quickly puncture the foil with instrument sampling probe, to a point about one-half of the headspace depth. Be careful that the probe does not pick up water droplets or soil particles.
5. Record the highest meter response as the jar headspace concentration. Maximum response should occur between 2 and 5 seconds after inserting the probe through the foil and into the jar.

Erratic meter response may occur when organic vapor concentrations are high or when excess moisture is present. These readings should not be used.

PID field instruments should be operated and calibrated to yield "total organic vapors" in ppm (v/v) as benzene. PID instruments must be operated with a 10.0 +/- eV lamp source. Operation, maintenance, and calibration should be performed in accordance with the manufacturer's specifications. For jar headspace analysis, instrument calibration should be checked and adjusted every 20 analyses (or each time the instrument is used, if less than 20 samples are analyzed).

F.7 Decontamination of Sampling Equipment

All non-disposable sampling equipment that comes in contact with the sample medium will be decontaminated to prevent cross-contamination between sampling points. This includes equipment such as stainless steel bowls, scoops, etc.

Standard Operating Procedures for decontamination procedures are addressed in the Tetra Tech SOP SA-7.1 (Appendix C). The following decontamination sequence will be employed:

- Remove gross contamination by scrubbing with potable water
- Scrub with potable water/liquinox
- Rinse with potable water
- Rinse with propanol
- Rinse with deionized water
- Air dry (to extent possible)
- Wrap with aluminum foil, dull side toward equipment.

APPENDIX G
LABORATORY SOPs



**LABORATORY
ACCREDITATION
BUREAU**

Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2247

MITKEM

175 Metro Center Blvd
Warwick, RI 02886

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: April 1, 2013

A handwritten signature in black ink, appearing to read 'R.D.L.', positioned above a horizontal line.

**R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 1st of April 2010**

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).



State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH

Certifies

LAI00301

MITKEM LABORATORIES A DIV OF SPECTRUM ANAL INC
UNIT 1
175 METRO CENTER BOULEVARD
WARWICK RI 02886-1755
Laboratory Director: KIN CHIU

for the analysis of: **Non-potable Water Organic Chemistry - Non-potable Water Inorganic Chemistry -**

This certification, pursuant to Rhode Island General Laws 23-16.2 supersedes, all previous Rhode Island certificates issued to this laboratory. Certification is no guarantee of the validity of the laboratory results.

This certificate is valid only when accompanied by the latest dated Appendix which lists the analytes and methods for which certification has been granted. Contact the Laboratory Certification Officer to verify the current certification status of this laboratory.

David R. Gifford, MD, MPH
Director of Health

Expires: 12/30/2009

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY IN A LOCATION VISABLE TO THE PUBLIC



State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH

APPENDIX TO ANALYTICAL LABORATORY CERTIFICATE No. LAI00301

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

Expiration Date: December 30, 2009

Issued: January 30, 2009

Non-Potable Water - Organic Chemistry

Acetone	EPA Method 624
Acetonitrile	EPA Method 624
Acrolein	EPA Method 624
Acrylonitrile	EPA Method 624
Benzene	EPA Method 624
Bromomethane	EPA Method 624
Bromoform	EPA Method 624
2-Butanone	EPA Method 624
Carbon Disulfide	EPA Method 624
Carbon Tetrachloride	EPA Method 624
Chlorobenzene	EPA Method 624
Chlorodibromomethane	EPA Method 624
Chloroethane	EPA Method 624
Chloromethane	EPA Method 624
2-Chloroethylvinyl Ether	EPA Method 624
Chloroform	EPA Method 624
Dichlorobromomethane	EPA Method 624
Dichlorodifluoromethane	EPA Method 624
Dibromomethane	EPA Method 624
1,2-Dibromo-3-chloropropane (DBCP)	EPA Method 624
1,2-Dibromoethane EDB	EPA Method 624
1,1-Dichloroethane	EPA Method 624
1,2-Dichloroethane	EPA Method 624
1,1-Dichloroethene	EPA Method 624
1,2-Dichloropropane	EPA Method 624
cis-1,3-Dichloropropene	EPA Method 624
trans-1,3-Dichloropropene	EPA Method 624
Ethylbenzene	EPA Method 624
Methylene Chloride	EPA Method 624
Hexachlorobutadiene	EPA Method 624
2-Hexanone	EPA Method 624
Naphthalene	EPA Method 624
Styrene	EPA Method 624
1,1,2,2-Tetrachloroethane	EPA Method 624
Tetrachloroethene	EPA Method 624
Toluene	EPA Method 624

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Issued: January 30, 2009

cis-1,2-Dichloroethene	EPA Method 624
trans-1,2-Dichloroethene	EPA Method 624
1,2,4-Trichlorobenzene	EPA Method 624
1,1,1-Trichloroethane	EPA Method 624
1,1,2-Trichloroethane	EPA Method 624
Trichloroethene	EPA Method 624
Trichlorofluoromethane	EPA Method 624
Vinyl Acetate	EPA Method 624
Vinyl Chloride	EPA Method 624
Xylenes (total)	EPA Method 624
1,2-Dichlorobenzene	EPA Method 624
1,3-Dichlorobenzene	EPA Method 624
1,4-Dichlorobenzene	EPA Method 624
MTBE	EPA Method 624
Acenaphthene	EPA Method 625
Acenaphthylene	EPA Method 625
Aniline	EPA Method 625
Anthracene	EPA Method 625
Benzidine	EPA Method 625
Benzo(a)anthracene	EPA Method 625
Benzo(a)pyrene	EPA Method 625
Benzo(b)fluoranthene	EPA Method 625
Benzo(k)fluoranthene	EPA Method 625
Benzo(g,h,i)perylene	EPA Method 625
Bis(2-Chloroethoxy)Methane	EPA Method 625
Bis(2-chloroethyl)Ether	EPA Method 625
Bis(2-chloroisopropyl) Ether	EPA Method 625
Bis(2-ethylhexyl) Phthalate	EPA Method 625
4-Bromophenyl Phenyl Ether	EPA Method 625
Butylbenzyl Phthalate	EPA Method 625
Carbazole	EPA Method 625
4-Chloroaniline	EPA Method 625
2-Chloronaphthalene	EPA Method 625
4-Chlorophenyl Phenyl Ether	EPA Method 625
Chrysene	EPA Method 625
Dibenz[a,h]anthracene	EPA Method 625
Dibenzofuran	EPA Method 625
1,2-Dichlorobenzene	EPA Method 625
1,3-Dichlorobenzene	EPA Method 625
1,4-Dichlorobenzene	EPA Method 625
3,3'-Dichlorobenzidine	EPA Method 625
Diethyl Phthalate	EPA Method 625
Dimethyl Phthalate	EPA Method 625
Di-n-butyl Phthalate	EPA Method 625
2,4-Dinitrotoluene	EPA Method 625
2,6-Dinitrotoluene	EPA Method 625
Di-n-octyl Phthalate	EPA Method 625
Fluoranthene	EPA Method 625



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Fluorene	EPA Method 625
Hexachlorobenzene	EPA Method 625
Hexachlorobutadiene	EPA Method 625
Hexachlorocyclopentadiene	EPA Method 625
Hexachloroethane	EPA Method 625
Indeno[1,2,3-cd]pyrene	EPA Method 625
Isophorone	EPA Method 625
2-Methylnaphthalene	EPA Method 625
Naphthalene	EPA Method 625
Nitrobenzene	EPA Method 625
2-Nitroaniline	EPA Method 625
3-Nitroaniline	EPA Method 625
4-Nitroaniline	EPA Method 625
N-Nitrosodimethylamine	EPA Method 625
N-Nitrosodi-n-propylamine	EPA Method 625
N-Nitrosodiphenylamine	EPA Method 625
Phenanthrene	EPA Method 625
Pyrene	EPA Method 625
Pyridine	EPA Method 625
1,2,4,5-Tetrachlorobenzene	EPA Method 625
1,2,4-Trichlorobenzene	EPA Method 625
2-Chlorophenol	EPA Method 625
2,4-Dichlorophenol	EPA Method 625
2,4-Dimethylphenol	EPA Method 625
2-Methyl-4,6-dinitrophenol	EPA Method 625
2,4-Dinitrophenol	EPA Method 625
2-Nitrophenol	EPA Method 625
4-Nitrophenol	EPA Method 625
4-Chloro-3-methylphenol	EPA Method 625
Pentachlorophenol	EPA Method 625
Phenol	EPA Method 625
2,4,5-Trichlorophenol	EPA Method 625
2,4,6-Trichlorophenol	EPA Method 625
Aldrin	EPA Method 608
alpha-BHC	EPA Method 608
beta-BHC	EPA Method 608
gamma-BHC (Lindane)	EPA Method 608
delta-BHC	EPA Method 608
alpha Chlordane	EPA Method 608
gamma Chlordane	EPA Method 608
Chlordane (technical)	EPA Method 608
4,4'-DDT	EPA Method 608
4,4'-DDE	EPA Method 608
4,4'-DDD	EPA Method 608
Dieldrin	EPA Method 608
Endosulfan I	EPA Method 608
Endosulfan II	EPA Method 608
Endosulfan Sulfate	EPA Method 608



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Endrin	EPA Method 608
Endrin Aldehyde	EPA Method 608
Heptachlor	EPA Method 608
Heptachlor Epoxide	EPA Method 608
Methoxychlor	EPA Method 608
PCB-1016	EPA Method 608
PCB-1221	EPA Method 608
PCB-1232	EPA Method 608
PCB-1242	EPA Method 608
PCB-1248	EPA Method 608
PCB-1254	EPA Method 608
PCB-1260	EPA Method 608
Toxaphane	EPA Method 608

Non-Potable Water - Inorganic Chemistry

Alkalinity	SM2320B
Aluminum	EPA Method 200.7
Ammonia	SM4500NH3C
Antimony	EPA Method 200.7
Arsenic	EPA Method 200.7
Barium	EPA Method 200.7
Beryllium	EPA Method 200.7
Boron	EPA Method 200.7
Bromide	EPA Method 300.0
Cadmium	EPA Method 200.7
Calcium	EPA Method 200.7
Chloride	EPA Method 300.0
Chloride	SM4500Cl E
Chromium (total)	EPA Method 200.7
Hexavalent chromium	SM 3500 Cr D
Cobalt	EPA Method 200.7
Copper	EPA Method 200.7
Specific Conductance	EPA Method 120.1
Fluoride	EPA Method 300.0
Hardness	SM2340B
Hardness	EPA Method 200.7
Iron	EPA Method 200.7
Lead	EPA Method 200.7
Magnesium	EPA Method 200.7
Manganese	EPA Method 200.7
Mercury	EPA Method 245.1
Molybdenum	EPA Method 200.7
Nickel	EPA Method 200.7
Nitrate	EPA Method 300.0
Nitrate/Nitrite as N	EPA Method 353.2
Nitrite	EPA Method 300.0



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Orthophosphate	EPA Method 300.0
Orthophosphate	SM4500 P E
Potassium	EPA Method 200.7
Selenium	EPA Method 200.7
Silver	EPA Method 200.7
Sodium	EPA Method 200.7
Sulfate	EPA Method 300.0
Sulfate	SM 426C 15h Ed.
Sulfide	SM 4500-S2-D
Thallium	EPA Method 200.7
Tin	EPA Method 200.7
Titanium	EPA Method 200.7
Vanadium	EPA Method 200.7
Zinc	EPA Method 200.7
Total Cyanide	EPA Method 335.4
Kjeldahl Nitrogen	SM4500N Org C
Oil & Grease	EPA Method 1664A
pH	SM4500HB
Total Organic Carbon	SM 5310B
Total Phenols	EPA Method 420.1
COD	SM5220D
Total Phosphorous	SM4500 P E
Total Suspended Solids	SM2540D
Total Dissolved Solids	SM2540C
Total Solids	SM2540B



**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

Rev. 5

Signature

Date

QA Director:

Sham B. Lawli

12/20/07

Lab Director:

Yuan D. J.

12/20/07

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

1. Scope and Application

This Standard Operating Procedure (SOP) describes the analysis of Semivolatile Polycyclic Aromatic Hydrocarbons (PAHs) in aqueous and solid sample using gas chromatography/mass spectrometry (GC/MS) and Selective Ion Monitoring (SIM). The SOP follows the analyses as discussed in current USEPA SW-846 Final Update IV , Method 8270D.

The semivolatile Polycyclic Aromatic Hydrocarbons that can be analyzed using this SOP are listed in **Table 1**. Additional SW-846 8270 semivolatile compounds can be analyzed by this method.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure and approve all reported results. The **Project Manager** evaluates laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

- 3.1 The samples are extracted using appropriate sample extraction methods (see SOPs for sample extraction) and, if necessary, sample clean-up procedures.
- 3.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer connected to the gas chromatograph.
- 3.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by coelution of the ions that are selected at the correct retention times. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum of a five-point calibration curve.
- 3.4 A list of acronyms used in this SOP is included in **Table 2**.

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers. For semivolatile organic compound analysis by the SIM method, water samples are collected in 1-liter amber glass bottles. Solid samples are collected in 8-ounce amber glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.
- 4.2 Sample extracts are transferred to the semi-volatile organics analysis lab with appropriate sample preparation information. Extracts are stored at $4^{\circ} \pm 2^{\circ}\text{C}$, protected from light, in sealed vials (screw-cap or crimp-cap) equipped with un-pierced PTFE-lined septa and stored in a separate location from the analytical standards.
- 4.3 The holding time of the extracts for semivolatile organic compounds analysis by the SIM method is 40 days from date of sample extraction. The sample preparation holding times are covered in the corresponding extraction procedures SOPs.

5. Interferences and Potential Problems

- 5.1 Evaluate the raw GC/MS data to verify that interferences were not introduced during the extraction and/or clean up of the samples.

- 5.2 The SIM method is not appropriate for multi-component analytes (Aroclor, toxaphene, chlordane, etc.). Refer to Methods 8081 and 8082.
- 5.3 This method is not appropriate for some of the routine SW8270 compounds. Semivolatile PAHs are routinely analyzed by the SIM method.

6. Equipment and Apparatus

6.1 Equipment:

- 6.1.1 There are five GC/MS in the semivolatile organic analysis lab. They are S1, S2, S3, S4 and S5. S1 and S2 have similar configurations, including Hewlett Packard (HP) Model 5890 gas chromatograph interfaced to HP Model 5972A mass spectrometer (GC/MS). S3 and S4 are composed of HP 6890 GC System (Model: G2614A) and Agilent 5973 MSD. S5 is a Hewlett Packard (HP) Model 7890A gas chromatograph with a front and rear injection port. The front injector is interfaced to HP Model 5975C mass spectrometer (GC/MS). The rear injector is interfaced with a flame ionization detector (FID). HP EnviroQuant Software is used to handle data acquisition. Target software by ThruPut is used for data reduction and report generation.
- 6.1.1.1 The HP GC is fitted with an electron pressure controller (EPC) to allow constant carrier gas flow during the temperature ramp.
- 6.1.1.2 A 30m x 0.25mm id (0.5 µm film thickness) DB-5MS fused-silica capillary column (J & W) is used for the analyses. Equivalent columns including HP-5MS have been used with similar performance.
- 6.1.1.3 A HP Model 6890 autosampler is used for sample injection on S1 and S2. An Agilent Autoinjection Tower 7683 is for S3 and S4. S5 is equipped with a CTC Analytical Leap autosampler capable of dual injections.

6.1.2 Instrument operating conditions are as follows:

General Gas Chromatography Conditions

Carrier Gas	Helium (99.999%)
Column Flow	about 1 mL/minute

Injector Temperature 290°C
Transfer Line Temperature 280°C
Injection Volume 1 µL

GC Temperature program

Ramp Rate	Initial Temp	Hold Time	Run Time
1	45°C	2.0 min	
2 15°C/min	225°C	0 min	
3 25°C/min	310°C	12.25min	29.65min

General Mass Spectrometry Conditions

Mass Range SIM scans specific to the masses for the individual compounds and surrogate.

Scan Speed at least 1 scan per second

Ionization Mode 70 eV positive ion

GC/MS program for DF TPP tune analysis:

DFTPP

GC Temperature program for analyzing DF TPP

Ramp Rate	Initial Temp	Hold Time	Run Time
1	150°C	1.0 min	
2 13 °C/min	300°C	2.0 min	14.54 min

GC/MS Program for Standards, Blanks, LCS, MS/MSD and sample analysis:

BNA_SIM_W or BNA_SIM_S, PAH_SIM_W or PAH_SIM_S

6.1.3 The MS program method scans for specific SIM masses in different time groups.

Group 1 scan should begin after the solvent delay and continue until the elution of 2-methylnaphthalene.

Group 2 scan should start right after Group 1 and continue until after fluorene.

Group 3 scan should begin right after Group 2 and continue until after anthracene.

Group 4 scan should begin right after *Group 3* and continue until after chrysene.

Group 5 should begin right after *Group 5* and continue until after benzo(g,h,i)perylene.

6.1.4 A primary and secondary ion for each analyte and surrogate will be collected. For some compounds an additional secondary ion will be collected to assist with identification. **Table 3** lists the primary and confirmation ions for target and surrogate analytes.

6.2 Preventative Maintenance - GC/MSs are maintained according to the manufacturer's recommendation. The lab analyst performs preventive maintenance as discussed below.

6.2.1 On a daily basis whenever analyses are to be performed, replace the GC septum and clean the injection liner. Also clip up to 6" of the column.

6.2.2 If needed, the analytical column is replaced; this is usually indicated by the tailing of the polar compounds such as pentachlorophenol/benzidine and/or initial and continuing calibration verifications that repeatedly fail to meet method requirements.

6.2.3 If the system constantly drifts out of DFTPP tune and/or the initial and continuing calibration verifications repeatedly fail to meet method requirements, the ion source will need to be cleaned.

6.2.4 There are two filaments in the mass spectrometer. If both filaments are blown, the method sample will be vented to replace both filaments.

NOTE: After major maintenance such as the scenarios described in **sections 6.2.2 through 6.2.4**, an Initial Calibration (ICAL) is analyzed. Document the date of the ICAL in the resolution field in the LIMS Maintenance Logbook.

6.2.5 The rough-pump oil will be replaced at least once a year. Check the oil level periodically and add oil if needed. Document this maintenance as above.

6.2.6 Once a year, all GC/MS systems may undergo extensive maintenance by a skilled technician. When this occurs, collect all associated paperwork and enter relevant information in the LIMS

maintenance log. The paperwork can be brought to the data reporting area for PDF inclusion on the server.

6.2.7 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the instrument vendor for telephone-consultation and if needed, schedule on-site repair. This information should be documented as in **Section 6.2.6**. In addition, the resolution field in the LIMS Maintenance Logbook should be filled in fully.

6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD hardware manual.

6.4 Glassware:

6.4.1 Hamilton syringes (10 μ l, 25 μ l, 100 μ l, 500 μ l, 1000 μ l). Syringe accuracy is certified to $\pm 1\%$ by the manufacturer.

6.4.2 Vials – 2ml glass with PTFE-lined screw-cap or crimp-cap tops.

7. Reagents

7.1 Organic solvents – J.T. Baker ultra analyzed grade methylene chloride for standard preparation

7.2 The standards used for this SOP are discussed below. ***Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*** These standards are obtained as ampulated mixture.

7.3 The laboratory will at all times archive or have on order one complete set of unopened ampulated standards (to include internal standards, surrogate standards and target analyte standards).

7.4 All **Primary** standards received from vendors are logged into the SVOA Primary Standard Logbook. The standards are labeled *SPyymmddX*,

where: SP = Semivolatle Primary Standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.4.1 Tune Standard: the tuning standard contains DF TPP, 4,4'-DDI, Pentachlorophenol and benzidine. It is purchased from Restek(Cat. No.31615) at 1000 ug/mL.

7.4.2 Internal Standard: the internal standard is obtained from Cambridge Isotope as neat compounds. A 2000ug/mL solution of all compounds (1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthalene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12) is prepared and transferred into 1mL aliquoted vials. An ampule is opened and then emptied into a mini-inert vial equipped with an on/off valve.

7.4.3 Primary Calibration Standard: the primary calibration standards are obtained from Restek :

- 8270 MEGA Mix (Restek, Cat. No. 31850) at 500-1000ug/mL

7.4.4 Second Source Standard: the second source standards are obtained from NSI:

- ICL BNA LCS Spike 100ug/mL (Cat No. WL-408-25)

7.4.5 Surrogate Standard: Benzo(e)pyrene d-12 standard is obtained from Cambridge (Cat.No DLM-257-S) at 200 ug/mL.

7.5 All **Intermediate** standards are logged into the SVOA Intermediate Standard Logbook. The standards are labeled *SIymmddX*,

where:

SI = Semivolatile Intermediate Standard

yymmdd = date the standard is prepared

X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.5.1 An intermediate 8270 standard is prepared by making a 10 times dilution of the primary standards in **Section 7.4.3**. An additional intermediate calibration standard at 10ug/mL is prepared by adding the following two standards, and diluting to 4 mL with methylene chloride.

8270 Intermediate (200 ppm)	200 uL
Benzo(e)pyrene-d12 (200 ppm)	200 uL

7.5.1.1 The Initial Calibration standards are prepared as follows:

Levels of	Volume (uL)	Volume (uL)
-----------	-------------	-------------

Initial Calibration (ug/mL)	Intermediate <u>Standard</u>	Methylene <u>Chloride</u>
10	1000	0
5.0	500	500
1 (L3)	100	900
0.5	50	950
0.1	10	990

7.5.2 The ICV is prepared in a similar manner as the Midpoint (L3) of the ICAL. Serial dilutions of the second source standards in **Section 7.4.4** are made until the result is a final 1ug/mL standard.

7.6 All **Working** standards are logged into the SVOA Working Standard Logbook. The standards are labeled *SWyymmddX*,

where: SW = Semivolatile Working Standard
yymmdd = date the standard is prepared.
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.6.1 The working tune standard at 50 ug/mL is prepared by adding 50uL of the stock standard to a final volume of 1 mL with methylene chloride.

7.6.2 The internal standard working standard at 500 ug/mL is prepared by adding 250uL of the primary standard (2000 ug/mL) and diluting it to 1 mL with methylene chloride.

Please note that the volumes can be adjusted to make larger or smaller volume of the standards.

The standards are protected from light and stored in the freezer (F7) at -10°C to -20° C. The standards are stored away from sample extracts to minimize cross contamination.

Unopened ampulated standards' expiration dates are based on manufacturer's expiration dates. If no manufacturer's expiration date is provided the ampulated standards may be retained unopened for up to two years. Once an ampulated standard is opened it may be retained for one year from the date it was opened. Intermediate and Working Standard expiration dates are up to 6 months after they are prepared.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8. Procedure

8.1 The SW-846 methods for sample extraction are as follows:

Method 3510 (SOP# 50.0051) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are serially extracted with methylene chloride using a separatory funnel. The final volume for the extracts is 0.5mL to achieve the lower reporting limit.

Method 3520 (SOP# 50.0050) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are placed in a continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0053) extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate, placed in an extraction thimble or between plugs of glass wool, and extracted using 1:1 v/v methylene chloride/acetone in a Soxhlet extractor.

Method 3550 (SOP# 50.0052) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate to form a free-flowing powder, then extracted by ultrasonic extraction using 1:1 v/v methylene chloride/acetone.

8.2 Tuning:

The tune standard is prepared at 50 µg/mL. The GC/MS must be tuned to meet decafluorotriphenylphosphine (DFTPP) criteria every 12 hours when standards, blanks, LCS and/or samples are to be analyzed.

All of the analysis information is to be recorded in the Semivolatiles Instrument Logbook. The logbook is issued by the QA officer and will be archived upon its completion.

8.2.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.1.1.4** to perform the tune analysis.

8.2.2 Acceptance criteria for tune - The DF TPP tune will be analyzed using scan for m/z of 35 to 500 amu, based on the full scan. The mass spectrum of DF TPP must be acquired in the following manner for analysis: three scans (the peak apex scan and the scans

immediately preceding and following the apex) are acquired and averaged; if needed, background subtraction will be performed and must be accomplished using no more than 20 scans prior to the elution of DF TPP. It is important that the analyst does not selectively add or subtract scans to generate the tune.

For SW846 projects, tune can also be obtained using one of the following procedures (1) use one scan at the peak apex, (2) scan immediately before or after the apex, (3) use the average across the entire peak

A typical mass spectrum and mass spectral listing of the tune in listed in **Figure 1**.

The acceptance criteria are as follows:

Mass	Ion Abundance
51	10 - 80% of Base Peak
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	10 - 80% of Base Peak
197	< 2.0% of mass 198
198	Base peak, or > 50% of mass 442
199	5.0 - 9.0% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present, < 24% of mass 442
442	Base Peak, or > 50% of mass 198
443	15 - 24% of mass 442

Once the mass spectrometer passes the DF TPP tune, all subsequent standards, samples, and blanks associated with the tune must be analyzed using identical mass spectrometer instrument conditions.

- 8.3 Initial Calibration - Initial calibration is performed after the instrument passes the tune, % breakdown requirements and column performance check. Initial calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial calibration will also be performed if continuing calibration analyses do not meet QA/QC criteria.

Five calibration standard solutions are required for all target and surrogate compounds. Standard concentrations of 10, 5.0, 1.0, 0.5, and 0.1 ng/ μ L are required for the surrogates and all compounds.

8.3.1 Calculation for Initial Calibration:

A typical chromatogram of a 2.0ng standard is shown in **Figure 2** attached with the quantitation report.

From the multi-level level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$\text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A_x = area of the selected ion for the target compound to be measured

A_{is} = area of the selected ion for the associated internal standard

C_{is} = concentration of the internal standard

C_x = concentration of the compound to be measured

The mean relative response factor is determined by averaging the 5 level values.

The % relative standard deviation (%RSD) of the RRF is also calculated using:

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

where: Standard Deviation = $\sqrt{\sum (X_i - X)^2 / (n-1)}$

where: X_i = each individual value used to calculate the mean

X = the mean of n values

n = the total number of values = 5

8.3.2 Initial calibration acceptance criteria for SW-846 is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of -50% to $+100\%$ of the mean area response of the internal standard in all of the calibration level.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the

mean retention time over the initial calibration range for each internal standard.

- The RSD for all target analytes and surrogate compound must be < 20%. The Target software will flag any compound whose RSD is greater than 20%. If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments

- (1) The method allows for a maximum of 10% of the target analytes and/or surrogate compound to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier RSDs have a maximum of 50%.
- (2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)

m = slope of the line

x = concentration of the calibration standard

b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin

The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$.

- (3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)

a and b = slope of the curve

x = concentration of the calibration standard

c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.3.3 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 1ug/mL standard prepared in **Section 7.5.2**. The acceptance criteria are as follows:

8.3.3.1 For routine SW8270 SIM analyses, the calculated value of the analyte in the ICV must be 70 – 130% of the expected value. DoD **full scan SW8270** analyses have an ICV recovery limit of 75-125%.

8.3.4 Initial calibration acceptance criteria must be met before any sample, blanks or LCS are to be analyzed.

8.3.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action includes preparing fresh standards, source cleaning, changing GC column or injection liners.

8.4 Continuing Calibration - Continuing calibration standards containing all of the target and surrogate compounds at 1 ng on-column injection is performed every time samples are to be analyzed to ensure that the GC/MS system continues to meet instrument sensitivity and linearity requirements.

8.4.1 Frequency of Continuing Calibration - The continuing calibration standard must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. The continuing calibration is also required whenever blanks, LCS, MS/MSD and samples are analyzed.

8.4.2 Continuing calibration acceptance criteria:

- The % D must be $\leq 20\%$ (use % drift if using a regression fit model). A maximum of 20% of the target analytes and/or surrogate compound are allowed to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier %Ds have a maximum of 50%.
- No quantitation ion may saturate the detector.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (1ug/mL) of the associated initial calibration.
- The internal standard area counts must be within +100% to -50% from that of the mid-point calibration (1ug/mL) of the associated initial calibration.

8.4.3 Corrective Action for Continuing Calibration - Depending on which compound(s) fail(s) the criteria, corrective action included preparing fresh standards, source cleaning, changing GC column or injection liners. Repeated failure to pass continuing calibration may necessitate performing new initial calibration.

8.4.4 Continuing calibration acceptance criteria must be met before any samples or blanks are to be analyzed for generation of acceptable data.

8.5 Sample Analysis:

Prior to sample analysis, the sample extract and the internal standard are allowed to warm to room temperature to ensure complete dissolution of the high molecular weight internal standards. Ten microliters of the internal standard solution is added to each of the sample extracts at 1.0 mL final volume to ensure 5.0ng on-column amount. Shake the extract slightly to mix. The internal standard volume will be adjusted for smaller extract volume.

8.5.1 Analytical Sequence: The following QC protocol is recommended for analyses:

Initial Batch	Middle/Final Batch
1. Tune including Breakdown	1. Tune
2. ICal Standard #1	2. %Breakdown
3. ICal Standard #2	3. CCV
4. ICal Standard #3	4. Method Blank
5. ICal Standard #4	5. LCS

- | | | | |
|--------|-------------------------------|--------|------------------------|
| 6. | ICal Standard #5 | 6-13. | Samples (< 8) |
| 7. | ICV | 14. | CCV |
| 8. | Method Blank | 15. | Method Blank |
| 9. | LCS | 16. | MS |
| 10. | LCSD | 17. | MSD |
| 11-18. | Samples (< 8) | 18-25. | Samples (< 8) |
| 19. | Tune (as required per 12 hr.) | 26. | CCV (Final Batch Only) |
| 20. | CCV (as required per 12 hr.) | | |
| 21. | Method Blank | | |
| 22. | MS | | |
| 23. | MSD | | |
| 24-31. | Samples (< 8) | | |

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative Retention Time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Coelution of the primary and confirmation ions.

9.2 Determining the Concentration of Target Compounds - Sample data should be reported in units of $\mu\text{g/L}$ for aqueous samples and $\mu\text{g/Kg}$ dry weight basis for solid samples.

9.2.1 Target Quantitation: Compounds identified are quantitated using the following equations:

$$\text{For aqueous samples, Concentration } \mu\text{g/L} = \frac{(A_x) (I_s) (V_t) (Df)}{(A_{is}) (RRF) (V_o) (V_i)}$$

$$\text{For solid samples, Concentration } \mu\text{g/Kg} = \frac{(A_x) (I_s) (V_t) (Df)}{(A_{is}) (RRF) (W_s) (V_i) (S)}$$

where: A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion of the associated internal standard

I_s = amount of internal standard added in nanogram

RRF = relative response factor

V_o = volume of water extracted in milliliters = 1,000

V_t = volume of the sample extract in milliliters = 1

Df = dilution factor

W_s = weight of soil extracted in grams

S = % solid

9.3 Rounding Rule – Use the most current EPA rounding rules.

9.4 Acceptance Criteria for Sample Analysis:

- the sample must meet both extraction and analysis holding time.
- the sample has to have a compliant tune, initial calibration and continuing calibration.
- the sample has to have a compliant method blank.
- the sample has to have a compliant LCS.
- the surrogate recovery per this SOP (**Section 10.5**) or client-specified criteria.
- all of the target analyte concentration should be within the calibration range.
- area count of each of the internal standards in the inclusive range of - 50% and +100% of the response of the continuing calibration
- retention time of each of the internal standards must not shift more than \pm 0.5 minute from the continuing calibration.
- excluding the solvent front or the aldol condensation peak for solid extract analysis, no ion should saturate the detector

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery } (\%R) = 100 \times (SSR-SR)/(SA)$$

where: SSR = spiked sample result

SR = sample concentration

SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results (IPANDA). To ensure the appropriate analyst is performing the analysis, the analyst's initials should be entered in the Enviroquant acquisition software (do not use the default value). The analyst processing and reviewing data will initial the Run Logbook when processing data.

10.2 Method blanks - A method blank is prepared and analyzed with every batch not to exceed 20 samples. Acceptance criteria for the method blank are as follows:

- The recovery of Benzo(e) pyrene-d12 must be within the acceptance limits discussed in **Section 10.5**.
- Method blank concentration for DoD full scan SW8270 projects must be less than one half the reporting limit.

10.2.1 The "B" qualifier is applied to positive sample results on Form 1 or LIMS Level 2 data sheet when the same compound is detected in the method blank.

10.3 Lab Control Sample (LCS) -- A Lab Control Sample is a weight or volume of a clean reference matrix (anhydrous sodium sulfate or DI water) that is spiked with target analytes and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 Acceptance criteria for LCS:

- General acceptance: compliant Benzo(e)pyrene-d12 recovery
- Recovery of individual compounds within 45-135% with the exception of pentachlorophenol which uses a 5-135% limit. These limits are currently being used per the ACOE guidelines until enough points are collected for both soil and water matrices, at which time in-house derived QC limits may be substituted.

10.3.2 If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged

with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

- 10.4 Duplicate Matrix Spikes: Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above. Control limits are the same as discussed in **Section 10.3** but are used as advisory guidelines.

If any compounds are outside of the acceptance limits, their recoveries and/or RPD are qualified with the "*" flag on the recovery MS/MSD summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries:

- 10.5.1 The recovery of Benzo(e)pyrene-d12 in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

- 10.5.2 Acceptance criteria - The percent recovery of benzo(e) pyrene in blanks, samples, duplicate matrix spikes and LCS must be within the set QC limits of 45-135%. These limits may be updated to in-house derived QC limits by the QA Department on an annual basis.

10.5.3 Corrective Action for Recovery Failures:

- 10.5.3.1 Any sample which fails the above will be subjected to re-extraction. Any method blank which fails the above will be re-extracted with the associated samples.
- 10.5.3.2 If re-extraction and re-analysis of the sample demonstrate similar recovery performance, both sets of results will be reported to demonstrate matrix-related problems. Re-extraction is not required if the recovery is out of the above range for both the native sample and its duplicate matrix spikes.
- 10.5.3.3 All surrogate outliers will be flagged with an "*" on the surrogate recovery report (Form 2) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports.

10.6 Internal Standard Response and Retention Times:

- 10.6.1 The area count of the characteristic ion of each of the internal standards in the samples, blanks, duplicate matrix spikes and LCS must be within the inclusive range of -50.0% and +100% of the response of the internal standards of the continuing calibration.
- 10.6.2 The retention shift for each of the internal standards in the samples, blanks and LCS must be within ± 0.5 minute of those obtained from the associated Continuing Calibration.

11. Data Validation and Reporting

- 11.1 All raw data, including calibrations, QC results, and samples results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory supervisor reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory.
- 11.2 Reports are generated by the reporting group. The data submitted for report preparation is dependent on project requirements and is subjected to further reviewed by the project manager for reasonableness prior to release to the customer.

12. Corrective Action Procedures

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy

report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedure SOP No. 80.0007. Starting in 2006, corrective actions have been recorded in the Omega LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

13. Health and Safety

- 13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.
- 13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan

15. References

U.S. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8270D, SW-846 Test Methods for Evaluating Solid Wastes, Update IV, Revision 4, February 2007.

Attachments:

- 1. Table 1:** PAH Target Analyte List For Method 8270 SIM
- 2. Table 2:** List of Acronyms.
- 3. Table 3:** Selected Ions for Target Compounds and Surrogate.
- 4. Figure 1:** DFIPP Tune and Chromatogram.
- 5. Figure 2:** Continuing Calibration Standard Chromatogram and Quantitation Report.

Table 1. PAH Target Analyte List For Method 8270 SIM

Target Analyte	CAS Registry No.
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-5
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
Phenanthrene	85-01-8
Pyene	129-00-0

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Table 2 – List of Acronyms

1,2-DCB	1,2-Dichlorobenzene
1,3-DCB	1,3-Dichlorobenzene
1,4-DCB	1,4-Dichlorobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
ACOE	Army Corp of Engineers
DoD	Department of Defense
RL	Reporting Limit
MDL	Method detection limit
MQL	Method quantitation limit
PQL	Project Quantitation limit
IS	Internal Standard
SS	Surrogate Standard
DFIPP	Decafluorotriphenylphosphine
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate

Table 3. Selected Ions for PAH Target Compounds and Surrogates

Target Analyte	Primary Quantitation Ion	Confirmation Ion
Acenaphthene	154	153
Acenaphthylene	152	151
Anthracene	178	89
Benzo(a)anthracene	228	114
Benzo(b)fluoranthene	252	126
Benzo(k)fluoranthene	252	126
Benzo(g,h,i)perylene	276	138
Benzo(a)pyrene	252	126
Chrysene	228	114
Dibenz(a,h)anthracene	278	139
Fluoranthene	202	101
Fluorene	166	165
Indeno(1,2,3-cd)pyrene	276	138
2-Methylnaphthalene	142	141
Naphthalene	128	129
Phenanthrene	178	89
Pyrene	202	101
<u>Surrogate:</u>		
Benzo(e)pyrene-d12	264	132

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Figure 1

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

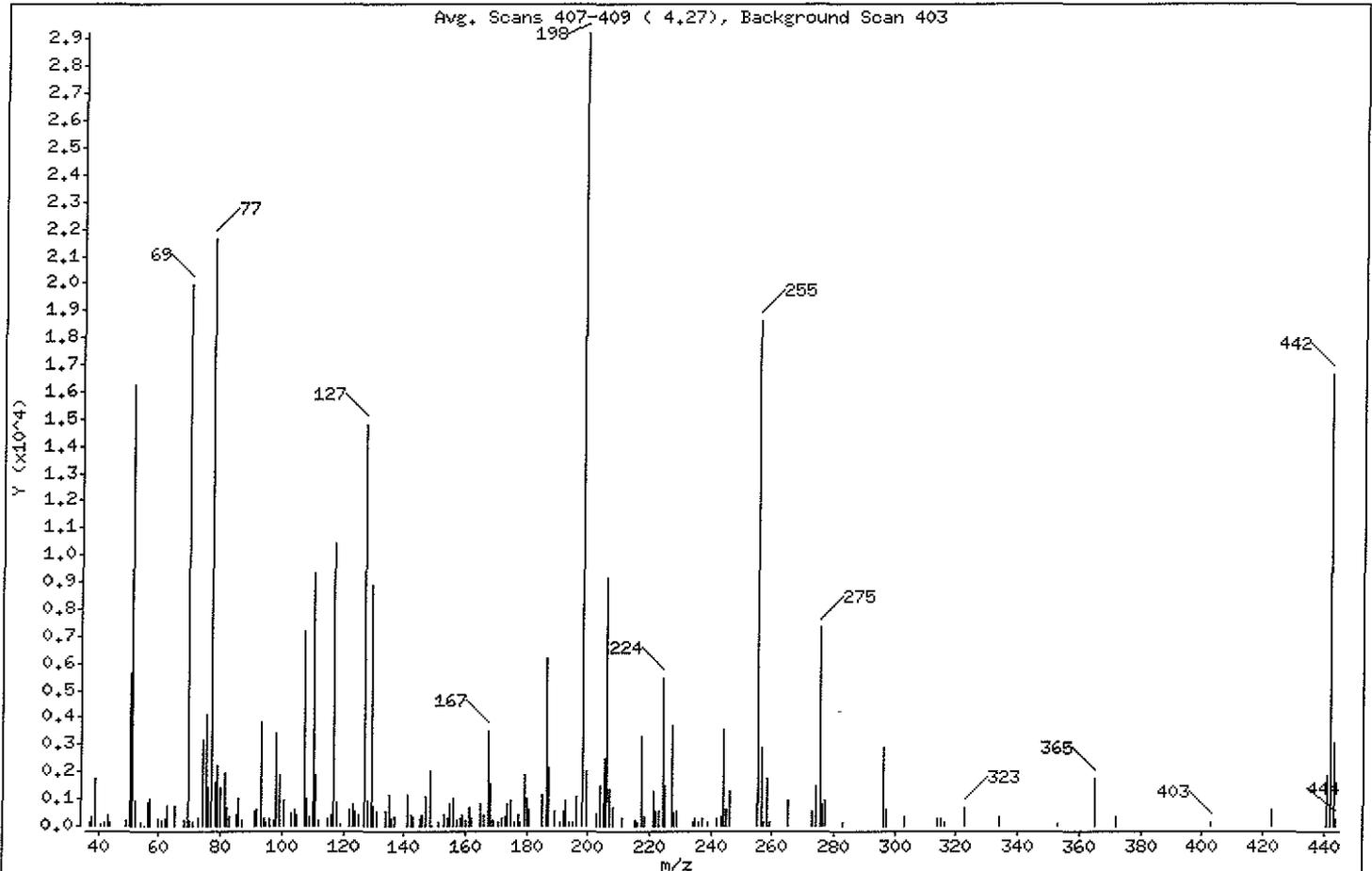
Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

1 dftpp



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	30.00 - 60.00% of mass 198	55.58
68	Less than 2.00% of mass 69	0.63 (0.93)
69	Mass 69 relative abundance	68.39
70	Less than 2.00% of mass 69	0.77 (1.13)
127	40.00 - 60.00% of mass 198	50.51
197	Less than 1.00% of mass 198	0.00
199	5.00 - 9.00% of mass 198	6.94
275	10.00 - 30.00% of mass 198	24.97
365	Greater than 1.00% of mass 198	5.87
441	Present, but less than mass 443	6.54
442	40.00 - 99.99% of mass 198	57.06
443	17.00 - 23.00% of mass 442	10.47 (18.35)

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0,25

Data File: S3E8260.D

Spectrum: Avg. Scans 407-409 (4,27), Background Scan 403

Location of Maximum: 198.00

Number of points: 180

m/z	Y	m/z	Y	m/z	Y	m/z	Y
37.00	126	99.00	1876	161.00	652	227.00	3694
38.00	345	101.00	957	162.00	263	228.00	455
39.00	1736	103.00	491	165.00	835	229.00	567
41.00	85	104.00	593	166.00	385	234.00	130
42.00	133	105.00	412	167.00	3471	235.00	236
43.00	431	107.00	7180	168.00	1531	236.00	101
44.00	113	108.00	1004	169.00	233	237.00	268
49.00	223	109.00	304	171.00	118	239.00	101
50.00	5664	110.00	9368	172.00	251	242.00	270
51.00	16247	111.00	1905	173.00	343	243.00	336
52.00	943	112.00	186	174.00	816	244.00	3547
54.00	146	115.00	273	175.00	919	245.00	607
55.00	27	116.00	436	176.00	147	246.00	1277
56.00	903	117.00	10447	177.00	391	255.00	18592
57.00	1024	118.00	885	178.00	126	256.00	2892
60.00	261	119.00	84	179.00	1906	257.00	117
61.00	209	122.00	631	180.00	1025	258.00	1759
62.00	262	123.00	836	181.00	623	259.00	167
63.00	734	124.00	513	185.00	1151	265.00	909
65.00	720	125.00	412	186.00	6167	273.00	518
68.00	185	127.00	14764	187.00	2152	274.00	1483
69.00	19992	128.00	948	189.00	533	275.00	7300
70.00	226	129.00	8882	191.00	115	276.00	837
71.00	112	130.00	752	192.00	530	277.00	958
73.00	286	131.00	565	193.00	920	283.00	132
74.00	3171	134.00	557	194.00	119	296.00	2884
75.00	4083	135.00	1134	195.00	102	297.00	625
76.00	1402	136.00	240	196.00	1047	303.00	317
77.00	21632	137.00	357	198.00	29232	314.00	250
78.00	1627	141.00	1174	199.00	2030	315.00	242
79.00	2243	142.00	425	203.00	490	316.00	101
80.00	1382	143.00	347	204.00	1470	323.00	682
81.00	1940	145.00	282	205.00	2453	334.00	318
82.00	694	146.00	409	206.00	9153	353.00	100
83.00	349	147.00	1046	207.00	1363	365.00	1717

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

Data File: S3E8260.D

Spectrum: Avg. Scans 407-409 (4.27), Background Scan 403

Location of Maximum: 198.00

Number of points: 180

m/z	Y	m/z	Y	m/z	Y	m/z	Y
85.00	412	148.00	2035	208.00	669	372.00	309
86.00	1023	149.00	22	211.00	293	403.00	132
87.00	201	151.00	109	215.00	228	423.00	575
91.00	526	153.00	434	216.00	137	441.00	1911
92.00	620	154.00	271	217.00	3296	442.00	16680
93.00	3798	155.00	776	218.00	309	443.00	3060
94.00	276	156.00	1027	221.00	1285	444.00	237
95.00	103	157.00	178	222.00	515		
96.00	269	158.00	284	223.00	564		
97.00	208	159.00	390	224.00	5411		
98.00	3434	160.00	190	225.00	1486		

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

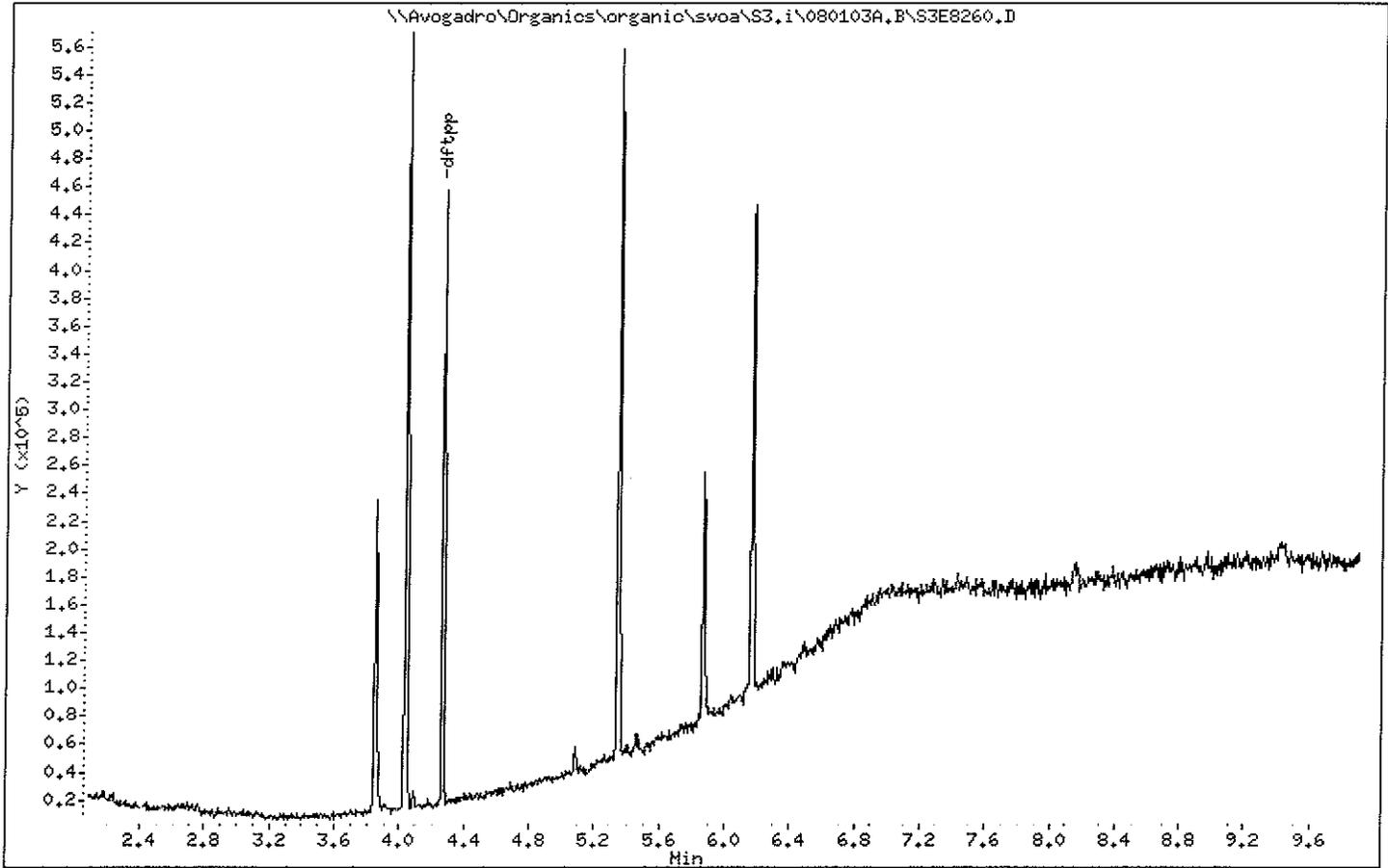


Figure 2

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8261.D

Date : 03-JAN-2008 17:57

Client ID: SSTD0013S

Sample Info: SSTD0013S,SSTD0013S

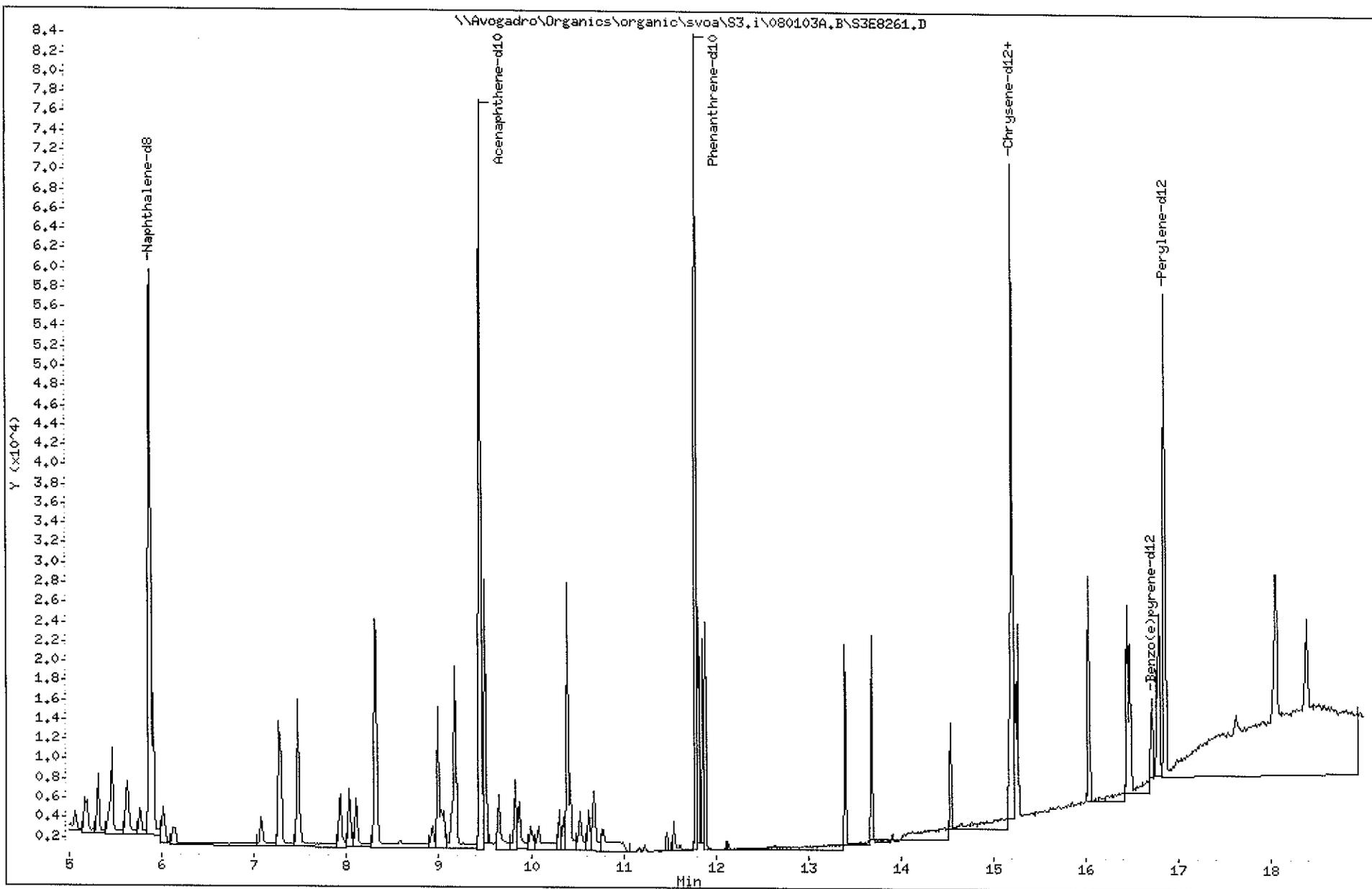
Volume Injected (uL): 1.0

Column phase: DB-5MS

Instrument: S3.i

Operator: CLM SRC: CLM

Column diameter: 0.25



Mitkem Corporation

SIM-PAH

Data file : \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8261.D
 Lab Smp Id: SSTD0013S Client Smp ID: SSTD0013S
 Inj Date : 03-JAN-2008 17:57
 Operator : CLM SRC: CLM Inst ID: S3.i
 Smp Info : SSTD0013S,SSTD0013S
 Misc Info : 2,3
 Comment :
 Method : \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\s3_pah_sim.m
 Meth Date : 14-Jan-2008 12:36 S3.i Quant Type: ISTD
 Cal Date : 28-DEC-2007 19:48 Cal File: S3E8155.D
 Als bottle: 1 Continuing Calibration Sample
 Dil Factor: 1.00000
 Integrator: HP RTE Compound Sublist: PAH.sub
 Target Version: 4.14

Concentration Formula: Amt * DF * Uf*(Vt/Vi)*(1/Vo) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	GPC Factor
Vt	1000.000	Extract Volume (uL)
Vi	1.000	Injection Volume
Vo	1000.000	Sample Volume
Cpnd Variable		Local Compound Variable

Compounds	QUANT SIG	MASS	RI	EXP RI	REL RI	RESPONSE	AMOUNIS	
							CAL-AMI (ng)	ON-COL (ng)
* 7 Naphthalene-d8		136	5.878	5.878	(1.000)	79435	5.00000	
8 Naphthalene		128	5.912	5.912	(1.006)	15041	1.00000	0.93
10 2-Methylnaphthalene		142	7.272	7.272	(1.237)	11217	1.00000	1.0
13 Acenaphthylene		152	9.169	9.169	(0.970)	20023	1.00000	0.89
* 14 Acenaphthene-d10		164	9.456	9.456	(1.000)	54655	5.00000	
15 Acenaphthene		154	9.504	9.504	(1.005)	12230	1.00000	0.91
18 Fluorene		166	10.397	10.397	(1.099)	15413	1.00000	0.95
* 23 Phenanthrene-d10		188	11.783	11.783	(1.000)	91276	5.00000	
24 Phenanthrene		178	11.814	11.814	(1.003)	22279	1.00000	0.89
26 Anthracene		178	11.886	11.886	(1.009)	21351	1.00000	0.88
27 Fluoranthene		202	13.397	13.397	(1.137)	20873	1.00000	0.85
28 Pyrene		202	13.681	13.681	(1.161)	22092	1.00000	0.62
30 Benzo(a)anthracene		228	15.187	15.187	(0.999)	19789	1.00000	0.94
* 31 Chrysene-d12		240	15.198	15.198	(1.000)	59780	5.00000	
32 Chrysene		228	15.230	15.230	(1.002)	18467	1.00000	0.96
33 Benzo(b)fluoranthene		252	16.445	16.445	(0.976)	17653	1.00000	0.86
34 Benzo(k)fluoranthene		252	16.477	16.477	(0.978)	18805	1.00000	0.98
\$ 35 Benzo(e)pyrene-d12		264	16.712	16.712	(0.992)	8469	1.00000	0.97
37 Benzo(a)pyrene		252	16.786	16.786	(0.996)	15758	1.00000	0.94
* 38 Perylene-d12		264	16.850	16.850	(1.000)	44148	5.00000	
39 Indeno(1,2,3-cd)pyrene		276	18.034	18.034	(1.070)	16569	1.00000	0.95

Data File: S3E8261.D
Report Date: 18-Jun-2008 09:33

Compounds	QUANT SIG		AMOUNTS				
	MASS	RI	EXP RI	REL RI	RESPONSE	CAI-AMI (ng)	ON-COI (ng)
===== 40 Dibenzo(a,h)anthracene	278	18 055	18 055	(1.071)	13767	1.00000	0.98
41 Benzo(g,h,i)perylene	276	18 375	18.375	(1.090)	14677	1.00000	1.00

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron

Capture Detector

Analysis by SW846 Method 8082

SOP No. 60.0003

Rev. 8

Signature

Date

QA Director:

Shamp B Lank

9/15/08

Lab Director:

C. J. Hart

9/15/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

**Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron
Capture Detector**

Analysis by SW846 Method 8082

Rev. 8

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the compounds listed in **Table 1**. This SOP describes the analysis of polychlorinated biphenyls (PCB) as Aroclors in solid or aqueous sample extracts, using a gas chromatograph equipped with an electron capture detector. This SOP gives specific information to perform the analysis according to protocols discussed in USEPA SW846 Test Methods for Evaluating Solid Waste Method 8082A, and Department of Defense Quality System Manual for Environmental Laboratories, Final Version 3. All matrices including ground water, aqueous samples, TCLP and SPLP extracts, petroleum oil, wipes, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require extraction and/or clean-up prior to analysis. **Section 8.2.1** provides the SOP references for sample extraction and clean-up procedures to be used with this analytical procedure. **Section 10** provides the quality control (QC) requirements required by Method 8082. A list of acronyms used in this SOP is included in **Table 2**.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors and Peer analysts** review the logbooks and data generated from this procedure and approve all reported results. The **Data Reviewer** evaluates all laboratory reports for reasonableness of the results. The **Project Manager** reviews the final report and signs the narrative. The **QA**

Director reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

- 3.1 A sample extract is analyzed by injecting a 1-2 μ L aliquot into a gas chromatograph (GC) using an auto-sampler. Concentrations of various PCB aroclors are determined by separation of the analytes using a GC equipped with fused silica, open-tubular, megabore columns and electron capture detectors (ECD). Hewlett Packard's Chem-Station (G1034C Version C03.00) is used to handle data acquisition. Target software from ThruPut (Revision 4.14) is used for data reduction and forms generation is through Omega LIMS.
- 3.2 While SW846 Method 8082 is also applicable to determining polychlorinated biphenyls as congeners, this SOP specifically analyzes PCB as aroclors.
- 3.3 PCB aroclors are determined using dual-column system with dissimilar phases. The method allows for the option of dual columns joined to a single injection port and individually connected to two ECDs

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are collected by clients and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients provide their own containers. For PCB analysis by USEPA SW846 Method 8082, water samples are collected in 1-liter amber glass bottles with no preservation added to the samples. Solid samples are collected in 8-ounce amber glass containers with no preservation needed. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.
- 4.2 All sample extracts are stored at 4°C \pm 2°C until analyzed.
- 4.3 Sample extract holding time for PCB analysis by the method is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOP.
- 4.4 The sample extracts are transferred from Organic Prep Laboratory with all appropriate sample prep information in 2ml auto-sampler vials with Teflon lined crimp cap. All vials should have a meniscus to mark the level of the extracts.
- 4.5. Samples, sample extracts and standards must be stored separately.

5. Interferences and Potential Problems

- 5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents, or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve the desired degree of discrimination and quantitation.
- 5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination. These materials may be removed prior to analysis using Gel Permeation Clean-up (Method 3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and batch analyzing the solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- 5.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. The solvent rinse should be followed by detergent wash with hot water and rinsed with tap water. Drain the glassware and dry overnight. Store dry glassware in a clean environment. Prior to use, rinse the glassware in methanol and methylene chloride respectively.
- 5.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCB aroclors. Sulfur contamination may be expected with sediment samples. Method 3660 is suggested for removal of sulfur.
- 5.5 Waxes, lipids, and other high molecular weight materials may interfere with the analysis of PCB aroclors and can be removed by Gel-Permeation Clean-up (GPC) (Method 3640). Co-eluting chlorophenols can be removed by florasil clean-up (Method 3620).

6. Equipment and Apparatus

- 6.1 Gas Chromatograph/Electron Capture Detector

There are 5 GC/ECD in the laboratory. Three of the instruments have similar configurations and are labeled as E1, E2 and E3. The other two are GC/ μ -ECD are labeled as E4 and E5. GC E1, E2 and E3 – Hewlett Packard (HP) Model 5890 Series II with electron Pressure Control. GC E4 and E5 - HP Model 6890 Series II with μ -Electron Pressure Control. All five GC's are temperature programmable instruments with single injection port and dual electron capture detectors, and are equipped with HP Model 7673A Auto-sampler. A single gooseneck splitless injection liner is used in the injection port and connected to a piece (up to a 5m long) of uncoated megabore column (0.53mm id) which serves as a guard column. A guard column is connected to the tee split. Each of two columns is connected to the split tee also. HP 3365 Chemstation software is used in conjunction with EnviroQuant software to handle data acquisition and processing. All GC's are interfaced to the network workstation. LIMS software is used for reports.

6.1.1 HP Vectra PC.

6.1.2 HP 3365, EnviroQuant and Target software.

6.1.3 HP Model 7673A auto-sampler.

6.1.4 Three pairs of chromatographic columns are intensively used in the laboratory:

Pair 1 DB-1701 column (J & W Scientific) 30 m x 0.53mm id (1.0 μ m film thickness) and DB-608 column (J & W Scientific) a 30 m x 0.53mm id (0.83 μ m film thickness)

Pair 2 RTX-CLPesticides (Restek) 30 m x 0.53 mm id (0.5 μ m film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.53 mm id (0.42 μ m film thickness)

Pair 3 RTX-CLPesticides (Restek) 30 m x 0.32 mm id (0.5 μ m film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.32 mm id(0.42 μ m film thickness).

6.1.5 Gooseneck splitless injection liner, Cat #20799 from Restek or equivalent

6.1.6 Uncoated megabore guard column (0.53 mm id, up to 5 m long). Cat # 10028 from Restek or equivalent.

6.1.7 Universal "Y" Press-tight tee split Cat # 20406 from Restek or equivalent.

6.2 Glassware:

6.2.1 Class "A" volumetric flasks:
10 ml, 25 ml, 50 ml, 100 ml, and 250 ml.

6.2.2 Syringes:
10 μ l, 25 μ l, 50 μ l, 100 μ l, 500 μ l, 1 ml, 2.5 ml (accuracy to \pm 1% per vendor's specification).

7. Reagents and Standards.

7.1 n-Hexane: Fisher Optima grade.

7.2 The standards used in the method are discussed below. Please note that standards from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials. The laboratory will at all time archive or have on order one complete set of un-opened ampulated standards.

7.3 The list of primary (ampulated) calibration standards are obtained from Restek:

- Aroclor 1016 and 1260 (Cat. # 32039) at 1000ug/mL.
- Aroclor 1221 (Cat.# 32007) at 1000ug/mL.
- Aroclor 1232 (Cat. # 32008) at 1000ug/mL.
- Aroclor 1242 (Cat. # 32009) at 1000ug/mL.
- Aroclor 1248 (Cat. # 32010) at 1000ug/mL.
- Aroclor 1254 (Cat. # 32011) at 1000ug/mL.
- Tetrachloro-m-xylene (TCX) (Cat.# 32027) at 200 ug/mL.
- Decachlorobiphenyl (DCB), (Cat.# 32029) at 200 ug/mL

7.4 The list of second source standards are obtained from Supelco:

- Aroclor 1016 (Cat. # 4-8097) at 1000ug/mL.
- Aroclor 1221 (Cat. # 4-8098) at 1000ug/mL.
- Aroclor 1232 (Cat. # 4-4805) at 1000ug/mL.
- Aroclor 1242 (Cat. # 4-4806) at 1000ug/mL.
- Aroclor 1248 (Cat. # 4-4807) at 1000ug/mL.
- Aroclor 1254 (Cat. # 4-4808) at 1000ug/mL.
- Aroclor 1260 (Cat. # 4-4809) at 1000ug/mL.

- 7.5 All of the above primary standards are logged into the Primary Pesticide Standard Logbook and labeled as **PPyymmddX**

Where: PP = Pesticides/PCB primary standard

yymmdd = date the standard is received

X = the order the standard is logged into the logbook on that date, in alphabetical order.

The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. All primary standards are stored according to manufacturer's recommendation. All vials containing primary standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability.

- 7.6 Intermediate Standards.

All intermediate standards are labeled **PIyymmddX** where:

PI = Pesticide Intermediate Standard.

yymmdd = date the intermediate standard is prepared.

X = the order that the intermediate standard is prepared on that date in alphabetical order.

Smaller or larger volumes may be used and the final volume adjusted to keep the concentration the same.

All intermediate standards are stored in amber glass bottles under refrigeration at 4°C or below. All standards are stored separately from samples and sample extracts

All intermediate standard solutions must be prepared every six months, or sooner, if the solution has degraded or concentrated.

All of the appropriate standard preparations are recorded in the Pesticide/PCB Intermediate Standard Preparation logbook.

Analyst must allow all intermediate standard solutions to equilibrate to room temperature before use.

- 7.6.1 Intermediate Pesticide/PCB Surrogate Mix:

The intermediate stock standard is made by diluting 0.5mL of stock TCX and 1.0 mL of stock DCB and diluting to 50 mL with hexane. Intermediate Pesticide/PCB Surrogate Mix contains TCX at 2 ug/mL and DCB at 4ug/mL.

7.6.2 Intermediate AR1660 (AR1016+AR1260) Stock Solution:

The two Aroclors –Aroclor1016 and Aroclor 1260 have different chromatographic patterns and can be combined for analysis. When the two Aroclors are combined, they are labeled as Aroclor 1660.

The mixture of the two Aroclors is prepared by diluting 1mL of primary Aroclor standard at 1000ug/mL (AR 1016 + AR1260) Mix into 100mLs of hexane.

Concentration of the intermediate AR1660 standard solution is 10ug/mL

7.6.3 Aroclors 1221, 1232, 1242, 1248 and 1254 Intermediate Stock Solutions:

Intermediate stock solutions of each of the aroclors are prepared by diluting 1mL of the primary standards (at 1000ug/mL concentration) to 100mLs using hexane. There will be one intermediate solution for each of the aroclors listed above, each at a concentration of 10µg/mL.

Please note that the following preparation procedures pertain to the use of the primary stock standards listed in **Sections 7.3** and **7.4**. Different preparation schemes are needed if different stocks are used.

7.7 Working Standards for Aroclor 1221, 1232, 1242,1248, 1254 and1660.

The working 5 levels calibration standards for each of the individual Aroclors is prepared as follows:

Level 5: 8mL of the intermediate stock Aroclor prepared in **Section 7.6.2**. and 2mL of the intermediate surrogate standard prepared in **Section 7.6.1**.are diluted to 50mL using hexane.

Note: The preparation volumes could be adjusted to make smaller or larger volume of standards as long as the final working concentrations are the same.

Level 4: 25mL of level 5 are diluted to 50mL using hexane.

Level 3: 25mL of level 4 are diluted to 50mL in hexane

Level 2: 25mL of level 3 standard is diluted to 50mL using hexane.

Level 1: 25mL of the level 2 standard are diluted to 50mL using hexane.

7.7.1 The 5 levels of Aroclor 1660 standards contain the following (concentration in ug/mL):

	Aroclor <u>1016</u>	Aroclor <u>1260</u>	<u>TCMX</u>	<u>DCB</u>
Level 1	<u>0.1</u>	<u>0.1</u>	<u>0.005</u>	<u>0.01</u>
Level 2	<u>0.2</u>	<u>0.2</u>	<u>0.01</u>	<u>0.02</u>
Level 3	<u>0.4</u>	<u>0.4</u>	<u>0.02</u>	<u>0.04</u>
Level 4	<u>0.8</u>	<u>0.8</u>	<u>0.04</u>	<u>0.08</u>
Level 5	<u>1.6</u>	<u>1.6</u>	<u>0.08</u>	<u>0.16</u>

7.7.2 The working 5 level of each of Aroclors contain the following (concentration in ug/ml):

	Aroclor	<u>TCMX</u>	<u>DCB</u>
Level 1	0.1	<u>0.005</u>	<u>0.01</u>
Level 2	<u>0.2</u>	0.01	<u>0.02</u>
Level 3	<u>0.4</u>	0.02	<u>0.04</u>
Level 4	<u>0.8</u>	<u>0.04</u>	<u>0.08</u>
Level 5	<u>1.6</u>	<u>0.08</u>	<u>0.16</u>

7.7.3 Second source standards:

The second source standards are obtained from Supelco. All Aroclors are individually prepared at 0.4 ug/mL.

Please note that surrogate standards may or may not be added to the second source standards.

All of the above working standards are logged into the Pesticide Working Standard Logbook and labeled as PWyyymmddX

where: PW = Pesticides/PCB working standard
 yyymmdd = date the standard is prepared
 X = the order the standard is prepared on that date,
 in alphabetical order

All working standards are stored in amber containers under refrigeration at 4°C or below. The standards are stored in separate location from the samples and/or extracts to minimize cross contamination.

The expiration date for the standards is six months from the date of preparation or whenever the primary standard expires, whichever comes first.

8. Procedure

8.1 Preparation – The methods in USEPA SW-846 for sample extraction are as follows:

Method 3510 (SOP# 50.0022) extracts aqueous samples for water-insoluble and slight water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

Method 3520 (SOP# 50.0025) extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0019) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using 1:1 (v/v) methylene chloride/acetone in a soxhlet extraction.

Method 3550 (SOP# 50.0016) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using ultrasonic extraction in 1:1 (v/v) methylene chloride/acetone.

Method 3570 (SOP# 50.0100) is the extraction of Pesticides in soil, sediment, tissues, biota and any sample considered solid. A 2-20gram sample is solvent extracted first with acetone, and then with hexane by either a manual shake approach or via rotation or spinning of the sample.

In addition, clean-up procedures are employed to remove co-eluting interferences. The lab uses GPC clean-up (Method 3640) and sulfur clean-up (Method 3660).

8.2 Instrument conditions

The GC operating conditions are as follows:

Carrier Gas	Helium (99.999%)
Column Flow	5-10 mL/min., independent of temperature
Make-up Gas	5% methane/95% argon (P-5)
Make-up Gas Flow	80-100 mL/min.

Injector	Split-less
Injector Temperature	200°C
Initial GC Temperature	<u>120-170°C</u>
Initial GC Hold	1 min.
Temperature Ramp	6-8°C/min
Final Temperature	265- <u>300°C</u>
Final GC Hold	2-15 min.
Detector Temperature	300°C

- 8.2.1 Optimize GC operating conditions for analytes separation and sensitivity. Once optimized the same conditions must be used for analysis of all standards, samples, blanks and MS/, MSD.

The auto-sampler makes 2µl injection for the analysis of all standards and sample extracts.

- 8.2.2 Preventative Maintenance.

The injector septum is replaced every time the instrument is set up to perform a sequence of analyses when a leak develops, or when initial and/or continuing calibrations fail to meet the method requirements.

The injection liner is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops or when initial and/or continuing calibrations fail to meet the method requirements. The gold seal will be replaced and the columns will be trimmed every time before a new calibration is run.

The column will be replaced if standard chromatograms show excessive peak tailing or initial and/or continuing calibrations repeatedly fail to meet the method requirements.

- 8.2.3 Major instrument maintenance must be documented in the LIMS maintenance logbook, regular (daily) maintenance can be documented in Instrument Run Logbook.

- 8.2.4 Initial calibration is performed by analyzing 5 level standards of the Aroclor 1660, and at least one level (midpoint) of the following:

- 1 Aroclor 1221
- 2 Aroclor 1232
3. Aroclor 1242
4. Aroclor 1248
5. Aroclor 1254
6. Aroclor 1660

All analytical runs need to be documented in the appropriate Instrument Run Log. In addition to listing the data file, the associated working standard ID and standard name should be noted.

The chromatograms for each of the target Aroclor standards are presented in **figures 1 - 7** for both the RTXCLPest and RTXCLPest II columns.

8.2.5 From the initial calibration, the following parameters are determined:

8.2.5.1 Retention time and retention time window:

The retention time window is established by evaluating the retention time of the Aroclor 1260 standards (at 1ug/mL) that were analyzed over a 72-hour interval. Please note that the laboratory does not need to perform retention time window studies for the other Aroclors.

From the standard analysis, three peaks are selected for each of Aroclor 1016 and Aroclor 1260. The retention time windows for the six peaks are determined as ± 3 times the standard deviation from the 72-hour interval. If the standard deviation is zero, a default standard deviation of 0.01 minutes will be used, per SW-846 8000C, Section 11.6.3.

The retention time windows are established by centering the ± 3 standard deviation around the retention time of the six peaks in the mid level standard that was analyzed as part of the 5 level initial calibration.

It is Mitkem's experience that the GC retention times operating under EPC conditions is very tight. Using the above approach in determining retention time windows at times has resulted in extremely narrow retention time windows. This may result in false negatives due to slight retention time shifts, especially when sample extracts are subjected to co-eluting interferences. In such cases, the SW-846 default standard deviations will be used.

For the surrogate standards, the retention time window is established using the same approach as the multi-component PCBs. The above retention time window criteria also apply.

8.2.5.2 Mitkem chooses to use external quantitation for instrument calibration and sample quantitation. The following equation is used to calculate the calibration factor (CF):

$$CF = \frac{\text{Peak area (or height) of standard}}{\text{Mass injected (ng)}}$$

The above equation is used to determine the calibration factor for both the single component and multi-component standards. Please note that either peak area or peak height can be used for the calculation.

The lab chooses to use peak height for single component analytes such as the surrogate compounds and for the multi-component PCB.

In calibrating for the Aroclors, the laboratory selects three peaks that are characteristic of each of the Aroclors. The analyst will choose peaks in each Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Also, it is preferable that one of the three peaks selected is unique to that Aroclor.

8.2.5.3 Linearity - linearity is used to evaluate the dynamic range of the calibration factor for each of the single component surrogate standards. Linearity, as measured by the % Relative Standard Deviation (RSD) is calculated using:

$$\% \text{ RSD} = \frac{SD_{CF}}{CF_{av}} \times 100$$

Where: $SD_{CF} = \sqrt{\sum (CF_i - CF_{av})^2 / (n-1)}$
 CF_{av} = average calibration factor
 CF_i = calibration factor
 n = total number of values = 5

Please note that there are three sets of calibration factors for each of the Aroclors.

8.2.5.4 %RSD Acceptance criteria for linearity:

The acceptance limit for linearity check is that the % RSD for the individual Aroclor calibration factors must be less than or equal to 20%. If this condition is met, the instrument is determined to be linear within the calibration range.

Given the large number of individual compound calibration factors, it is likely that some Aroclors may exceed the 20%

acceptance limit. For Aroclors that exceeded the 20% limit, the linearity check is still acceptable if the following least square regression conditions are met:

8.2.5.5 Alternate Least Square Regression criteria for Linearity:

The analyst may employ a regression equation for the Aroclor(s) that do(es) not pass using the earlier approach. The regression will produce the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where y = instrument response (peak area or height)

m = slope of the line

x = concentration of the calibration standard

b = intercept

It is important that the origin (0,0) is not included as the Sixth calibration point and that the above equation is not forced through the origin. The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$ ($r^2 \geq 0.990$)

- 8.2.6 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. The calibration is performed by analyzing standard Aroclor 1660 standard prepared in **Section 8.1.8**. The acceptance criteria are as follows:

The calculated value of the analyte in the ICV should be 80 – 120% of the expected value. If the ICV does not meet the criteria, see corrective action guidelines in **Attachment 1** of this SOP. Results of this evaluation should be documented in the Instrument run log.

If the above criteria are not met, the analyst must evaluate the integrity of the primary and confirmation standards. If needed, re-preparation and re-analysis of the initial calibration is required.

- 8.2.7 Initial calibration acceptance criteria must be met before any sample, blanks or LCS are to be analyzed.
- 8.2.8 Corrective Action for Initial Calibration – see **Attachment 1** for corrective action guidelines and documentation.

8.2.9 Original initial calibration raw data must be archived in the company organics analysis calibration (OCAL) database. See Mitkem SOP # 10.0009 (Application Xtender) for scanned archive information.

8.3 Continuing Calibration Verification and Sample Analysis – Method 8082 requires the analysis of Aroclors 1016/1260 as the continuing calibration verification (CCV) every time samples are to be analyzed to ensure that the GC/ECD system continues to meet instrument sensitivity and linearity requirements. Mitkem may also analyze Aroclors 1242, 1248 and 1254 as part of the calibration verification

8.3.1 Frequency of Continuing Calibration (CCV)- The CCV must be performed if the instrument has been idle for more than 12 hours. A continuing calibration standard must also be injected at intervals of not less than once every twenty field samples (the method recommends the frequency to be once every ten samples, to minimize reanalyses due to unacceptable CCVs) and at the end of the analysis sequence.

DoD requires CCV standards must be injected at intervals of not less than once every ten field samples.

8.4.1 The continuing calibration is performed using the Level 3 (0.4 ug/ml) Aroclor 1660 standard (as well as Aroclors 1242, 1248 and 1254 where appropriate). While the method suggests alternating the use of high and low calibration mixtures, Mitkem performs the continuing calibration using the mid-point standard. When the additional Aroclors 1242, 1248 and 1254 are analyzed, they will also be evaluated using the same approach as Aroclors 1016/1260.

8.3.2 Evaluation of the Calibration Verification:

8.3.2.1 *% Difference*: Calculate the % difference between the continuing calibration CF and those from the most recent initial calibration.

The % difference is determined as follow:

$$\% \text{ Difference} = \frac{CF_c - CF_i}{CF_i} \times 100$$

In which:

CF_c = calibration factor from continuing calibration
CF_i = mean calibration factor from the most recent initial calibration

8.3.2.2 *% Drift*: Use % drift when using linear regression calibration.

$$\% \text{ Drift} = \frac{\text{Conc}_c - \text{Conc}_t}{\text{Conc}_t} \times 100$$

In which: Conc_c = concentration obtained from continuing calibration

Conc_t = theoretical concentration of standard

8.3.2.3 *% Difference (or % Drift) between the continuing calibration factor and that of the initial calibration should be no greater than ± 20% at least on one column. If this criterion is met then the calibration has been verified and sample analysis can proceed. The analyst should mark whether the CCV passed on one or both columns in the instrument logbook. Checkmarks or the term "OK" are acceptable entries.*

8.3.2.4 Comparison of the retention time window: the retention time of each aroclor and surrogate peak in the calibration standards should fall within the retention time window established from the initial calibration. Due to the need to perform routine column maintenance (clipping off a small length of the guard and/or analytical columns), the retention time might be shortened. In such cases, either the RT window is adjusted to agree with the calibration verification retention times or new retention times are established using the calibration verification standard.

8.3.3 Corrective Action for opening CCV- The %D (or % Drift) between the individual compound calibration verification factors and those from the initial calibration should be no greater than ±20% on at least one column. If this criterion is exceeded for any peak, the calibration has not been verified.

If the continuing calibration does not meet the criteria, a second injection of the calibration verification is allowed. If the second injection fails, the analytical sequence is stopped. Corrective actions must be performed. This may include preparing new standards, performing inlet maintenance and/or GC column maintenance. If the response of the analyte is still not within the above criteria, then a new initial calibration must be performed.

8.3.4 Corrective Action for closing CCV: When a calibration verification standard fails to meet the QC criteria on both columns, all samples that were injected after the last compliant standard must be evaluated to ensure the data is valid. The analyte(s) which fail(s) the 20%D must be addressed if the analyte affected is one that was detected in project sample(s). The analyst needs to note all QC issues in the instrument run

log and on all associated project data review checklists. This information needs to be addressed in the associated project(s) narrative. See **Attachment 1** for corrective action guidelines and documentation.

8.3.4.1 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (positive bias), and the analyte **was not** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, the group of samples is deemed acceptable and does not require re-analysis.

8.3.4.2 If the non-compliant standard analyzed after a group of samples exhibits an elevated response for an analyte that is greater than 20% D (positive bias) on both columns, and the analyte was detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, then re-injection of all samples are required to ensure accurate quantitation.

8.3.4.3 If the non-compliant standard analyzed after a group of samples exhibits an elevated response for an analyte that is greater than 20% D (positive bias) on one column, and the analyte was detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, and the associated samples are detected, the reported values must come from the compliant column, even if this is the higher result.

8.3.4.4 If the non-compliant standard analyzed **after** a group of samples exhibits a lower response for an analyte that is greater than 20% D (negative bias), then re-injection of all samples are required to ensure accurate quantitation.

Sample injections may continue as long as the calibration verification standards meet the instrument QC requirements.

All standard analyses are to be documented in the appropriate Instrument Run Log with associated working standard IDs.

8.3.5 The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Target software. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample. Both retention time and pattern are important when determining PCBs in a sample.

It is important that if Aroclors are detected in the samples, the analysis of these sample extracts should be accompanied by the appropriate Aroclor calibration verification standard as part of the analytical sequence. If this is not done, the sample extracts have to be re-injected with the appropriate Aroclor standards to ensure the PCB pattern is similar.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. In these instances, the analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclors.

8.3.6 The concentration of the target compounds and surrogates are calculated separately for each of the three Aroclor peaks for both columns using the following equation.

$$\text{For aqueous samples, concentration } \mu\text{g/L} = \frac{(\text{Ax}) (\text{Vt}) (\text{DF})}{(\text{CF}) (\text{Vo}) (\text{Vi})}$$

$$\text{For soil samples, concentration } \mu\text{g/kg} = \frac{(\text{Ax}) (\text{Vt}) (\text{DF})}{(\text{CF}) (\text{Ws}) (\text{Vi}) (\text{S})}$$

in which: Ax = area/height of the peak for the compound to be determined

CF = calibration factor

Vo = volume of water extracted, in mL

Ws = weight of soil sample, in gram

Vi = volume of extract injected, in μL

Vt = volume of extract, in μL

DF = dilution factor

S = solid content, expressed in decimal

For the multi-component Aroclors, the analyte concentration is calculated by taking the average of the concentration determined for each of the three peaks selected for that analyte.

- 8.3.7 Manual integration will be performed if needed and documented according to the current revision of SOP Number 110.0008. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. The analyst will further initial and date the manual integration with the proper reason code per SOP No.110.0008.
- 8.3.8 The concentrations of the surrogate analytes are calculated and reported separately for each of the two columns. The acceptance limits for the recoveries are discussed in **Section 10**.
- 8.3.9 The analyte recoveries of the LCS and matrix spike compounds are determined and reported. The acceptance limits for the recoveries are discussed in **Section 10**.

8.4 Analytical sequences are summarized as follows:

Order	Sequence	Comment
1	AR1232L3	
2	<u>AR1221L3</u>	
3	AR1242L1	Level 3 only for AR1232 <u>and</u> <u>AR1221</u> are run unless this Aroclor is detected in samples.
4	AR1242L2	
5	AR1242L3	
6	AR1242L4	
7	AR1242L5	
8	AR1248L1	
9	AR1248L2	
10	AR1248L3	
11	AR1248L4	
12	AR1248L5	
13	<u>AR1254L1</u>	
14	<u>AR1254L2</u>	
15	<u>AR1254L3</u>	
16	<u>AR1254L4</u>	
17	<u>AR1254L5</u>	
18	AR1660L1	
19	AR1660L2	
20	AR1660L3	
<u>21</u>	AR1660L4	
<u>22</u>	AR1660L5	
22	AR1660SS	Second source <u>ICV</u>

23	BIBLK01	Instrument blank
24	Blank	
25	LCS	
26	Sample extracts	
27	AR1242M01	<u>calibration verification</u>
28	AR1248M01	
29	<u>AR1254M01</u>	
30	AR1660M01	
31	BIBLK02	
32	Sample extracts	
33	AR1242M02	
34	AR1248M02	
35	<u>AR1254M02</u>	
36	AR1660M02	
37	BIBLK03	
38...	Sample extracts...	Sequence continues as the same as above as long as <u>calibration verifications</u> meet the acceptance criteria.

9. Data Reduction and Calculations

- 9.1 Sample data should be reported in units of $\mu\text{g/L}$ for aqueous samples and $\mu\text{g/Kg}$ dry weight for solid samples.

Results are reported to two significant figures using the USEPA guidelines in rounding up or down. For solid samples, results are reported in dry weight unless otherwise specified.

For 8082 analyses, PCB aroclors are reported if the determined concentration is above the project reporting limits on both columns. No J flagged results are reported.

- 9.2 Soil concentrations are calculated using dry weight basis. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids.

$$\% \text{ solids (S)} = \frac{\text{DW}}{\text{WW}} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
 WW = Sample weight (g) before drying

- 9.3 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100\% \times (\text{SSR} - \text{SR}) / (\text{SA})$$

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

- 9.4 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100\%$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

- 10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results.
- 10.2 Method blanks. A method blank is an aliquot of a clean reference matrix (reagent water for water samples, or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. A Method blank is prepared and analyzed with every batch of 20 samples or less. It is used to determine the level of contamination associated with the analytical processing and analysis of the samples. The Method blank **MUST** be analyzed on the same instrument as the associated samples.
- See **Attachment 1** for additional corrective action.
- 10.2.1 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.
- 10.2.2 The concentration of target compounds in the method blank must be \leq to $\frac{1}{2}$ CRDL or RL.
- 10.2.3 Corrective action for method blank contamination involves determining the source of the contamination and possibly re-extracting the entire batch.
- 10.3 Lab Control Sample (LCS) – A Lab Control Sample is a volume or weight of a clean reference matrix (organic-free water or Ottawa Sand) that is spiked with Aroclors 1016 and 1260 and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

- 10.3.1 The recovery of the surrogates must be within the calculated acceptance limits discussed **Section 10.6**.
- 10.3.2 The LCS recovery is evaluated against the established in-house limits. Any analyte/analytes not meeting the criteria require corrective action. See **Attachment 1**. For DoD projects, see **Attachment 2**, DoD Tables D-14 and 15.
- 10.3.3 Advisory RPD limits are set at 30% when duplicate LCSs are performed.

Any sample(s) that is/are associated with a non-compliant LCS may require re-extraction and re-analysis. See **Attachment 1** for corrective action guidelines.

- 10.4 Duplicate Matrix Spikes –Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix at a frequency of one set per 20 samples.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and one duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform a matrix spike and matrix spike duplicate.

- 10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above.

- 10.4.2 Control limits for Method 8082 projects are the same as discussed in **Sections 10.3.2 and 10.3.3**. DoD requires 30% RPD for all matrices. See **Attachment 2**, DoD Table B-2.

Depending on sample matrix, chromatographic based analysis such as Method 8082 is susceptible to co-eluting interferences resulted in high biased values. Thus, the control limits for the duplicate matrix spikes should be used as guidelines. The determination of outliers should be addressed in the project narrative to advise the data users of potential matrix related interferences.

- 10.5 Re-analysis at dilution - Any target compounds that are determined above the instrument calibration range will require reanalysis at dilution. A notation for the need to perform a dilution next to the sample run in the instrument logbook is needed to document the results. An entry such as “ rerun at 4x ” is acceptable.

The dilution is performed by taking an aliquot of the extract and diluting it to a pre-determined volume using hexane. The analyst should not over-dilute the extract. The analytes that trigger the need for dilution should be determined in the diluted analysis at a concentration above that of the midpoint calibration standard.

10.5.1 When reporting diluted results the following guidelines should be followed. If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis may be reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the DL suffix appended to both the client and sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and all compounds detected in the more diluted (DL) analysis are qualified with the "D" flag.

10.5.2 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument, the initial analysis may be performed at dilution. If this analysis is acceptable (compounds at or above the mid-point calibration) then a less dilute analysis is not required. The sample ID is not changed by adding a DL suffix but the 'initial analysis at dilution' is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

10.5.3 If the initial analysis fails QC criteria, it may be reported if specified by the project or client. If only the dilution is reported, the DL suffix is not added and the dilution is noted on the data review checklist submitted with the data for review as above.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks, MS/MSD and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1 Acceptance criteria for surrogate recovery.

For 8082 projects the percent recovery of each of the surrogate compounds in method blanks and LCS must be within the in-house acceptance criteria window. The current in-house acceptance criteria for each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS are listed under the option SPEC for the Omega LIMS Testcode.

In-house limits may be lab generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab generated limits are derived, these will be updated annually.

Surrogate spikes in matrix specific samples that fail to meet the in-house limits would indicate potential matrix effect. In general, high recovery of surrogates due to co-eluting interferences is acceptable. Low recovery of surrogates would indicate potential matrix related problems or one related to the extraction process. The analyst should confirm the sample volume extracted and the final extract volume to verify the calculations. If applicable, the analyst should consider additional cleanup procedures and re-analysis. If no appropriate cleanup procedure could be identified or re-analysis after additional cleanups failed to achieve the limit, this should be noted on the review checklist and also addressed in the narrative.

If surrogate recoveries do not meet recovery criteria see **Attachment 1** for corrective action

- 10.7 Annually, MDL studies are conducted to establish the limit of detection applicable to this method. Please refer to the SOP No. 80.0005 for more detail. An MDL verification at approximately 2XMDL will be analyzed after the study. MDL verification may be analyzed quarterly in lieu of the annual study.

11. Data Validation and Reporting

- 11.1 All raw data, including calibrations, QC results, and samples results, is reviewed for technical accuracy and completeness by the analyst. The analyst initiates a project data review checklist and documents all comments regarding analysis there. Sample preparation logs, notebooks, and instrument logs are reviewed and signed by the laboratory supervisor. The QA Director randomly reviews 10% of the data reported by the laboratory.
- 11.2 All raw data is peer reviewed at the computer/Target level by another analyst or the lab supervisor prior to final form generation. Analysts generate all hard copy raw data and upload electronic data files to the OMEGA LIMS for reporting. Raw Data, including all support data (such as data review checklist, run-logs, work-order sheets, SDG summaries...) are brought to the data reporting department for assembly. After assembly, all data are reviewed by senior personnel (data reviewers) for quality control and completeness dependent on project specific requirements.

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. See **Attachment 1** for routine corrective action guidelines and documentation.

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedures SOP No. 80.0007.

13. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible. Spent vials and ampules are disposed of into a red metal drum in the Semivolatle Laboratory. See SOP No. 30.0024, Sample and Waste Disposal for more detail.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention; Waste Management; Definitions and Acronyms

See Sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency. Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Method 8082A, SW-846 Test Methods for Evaluating Solid Wastes, Final Update IV, Feb 2007.
2. "Methods Compendium for Inorganic and Organic Methods," United States Army Corps of Engineers, Appendix I, 2001.
3. "Shell for Chemical Analytical Requirements," United States Army Corps of Engineers, Appendix H, 1997 including addendum dated 1 February 2001.
4. Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

Attachments:

1. **Table 1:** List of Target Analytes.
2. **Table 2:** List of Acronyms.
3. **Figure 1:** Aroclor 1660 Standard Chromatogram and Quantitation Report.
4. **Figure 2:** Aroclor 1221 Standard Chromatogram and Quantitation Report.

5. **Figure 3:** Aroclor 1232 Standard Chromatogram and Quantitation Report
6. **Figure 4:** Aroclor 1242 Standard Chromatogram and Quantitation Report
7. **Figure 5:** Aroclor 1248 Standard Chromatogram and Quantitation Report
8. **Figure 6:** Aroclor 1254 Standard Chromatogram and Quantitation Report
9. **Attachment 1:** Corrective Action Examples and Documentation Tables
10. **Attachment 2:** DoD QC Requirements: SW 846 DoD-B, Tables B-2, D-14, D-15.

Table 1**List of Target Analytes for Method 8082**

<u>Aroclor</u>	<u>CAS Number</u>
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

Table 2**List of Acronyms**

PCB	Polychlorinated Biphenyl
TCLP	Toxicity Characteristic of the Leaching Procedure
SPLP	Synthetic Precipitate Leaching Procedure
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate
GC	Gas chromatograph
ECD	Electron capture detector
GPC	Gel Permeation chromatography
CF	Calibration factor
RSD	Relative standard deviation
RT	Retention Time
EPC	Electron pressure controller
USACE	US Army Corp. of Engineers
AFCEE	Air Force Center of Environmental Excellence
CLP	US EPA Contract Laboratory Program
DoD	Department of Defense

Figure 1

Aroclor 1660 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D

Date : 31-MAR-2006 02:31

Client ID: AR1660FL1

Sample Info: AR1660FL1,AR1660FL1,,ar1660.sub,,

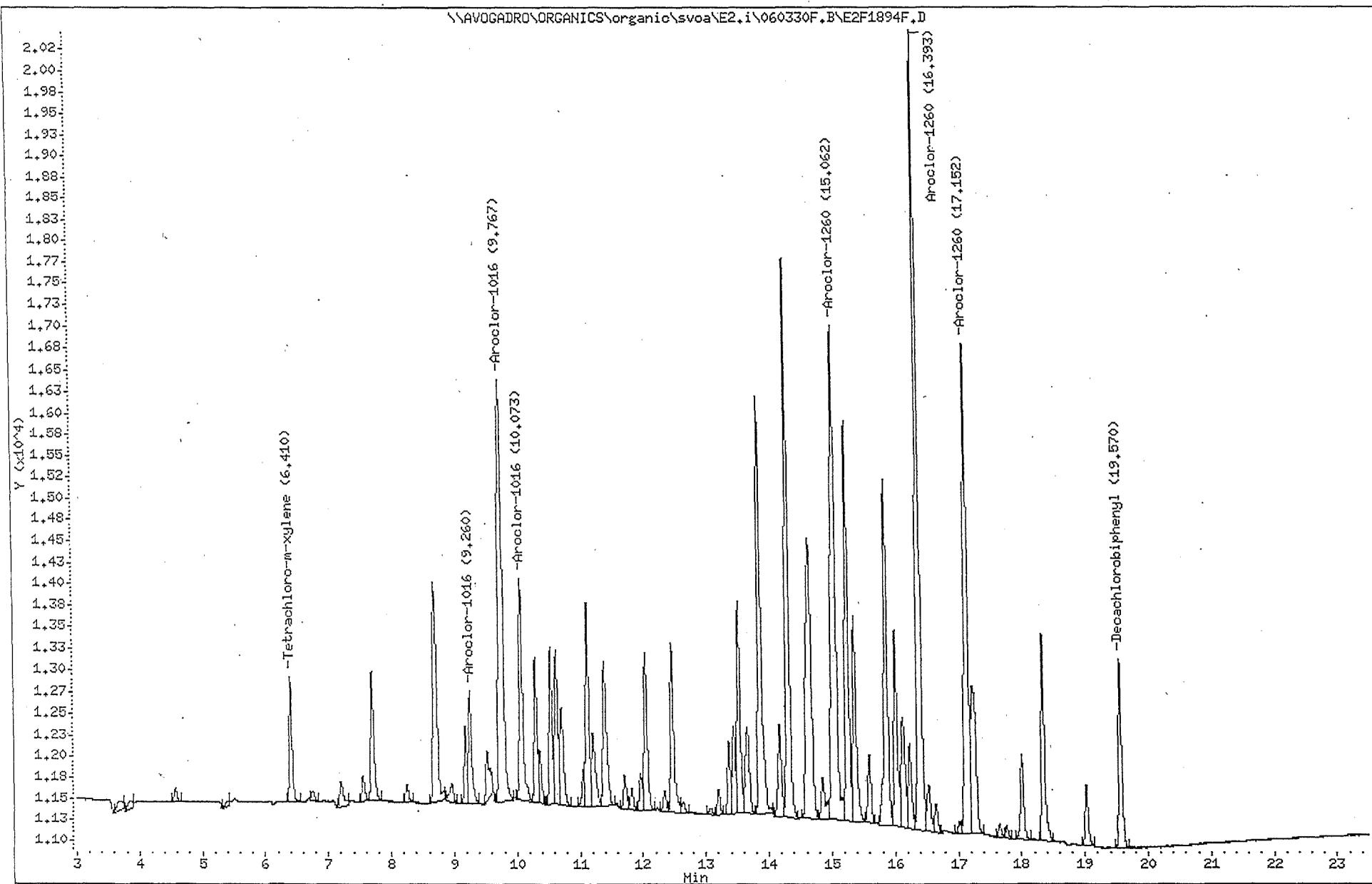
Volume Injected (uL): 1.0

Column phase: RtxCLPPest

Instrument: E2,i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D

Date : 31-MAR-2006 02:31

Client ID: AR1660FL1

Sample Info: AR1660FL1,AR1660FL1,,ar1660,sub,,

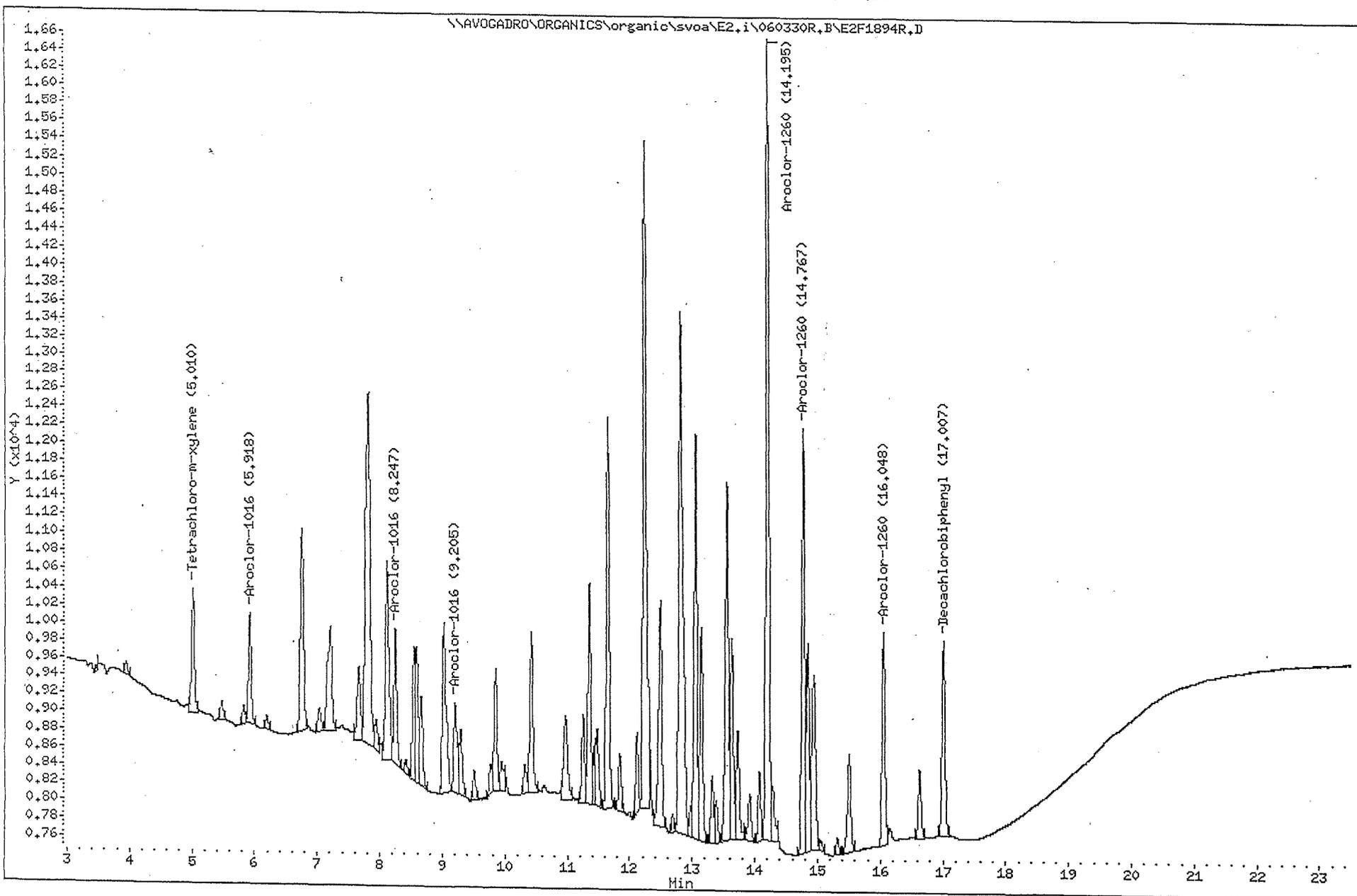
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1894F.D
 Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D
 Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
 Inj Date : 31-MAR-2006 02:31
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 18 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1660.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1 Tetrachloro-m-xylene			CAS #: 877-09-8			
6.41	6.41	0.000	1471 0.00200	0.0022		(a)

4 Aroclor-1016			CAS #: 12674-11-2			
9.26	9.26	0.000	1314 0.10000	0.12	80.00- 120.00	100.00(a)
9.77	9.77	0.000	4921 0.10000	0.13	327.41- 367.41	374.51
10.1	10.1	0.000	2590 0.10000	0.12	166.80- 206.80	197.11
Average of Peak Amounts =			0.12			

\$ 10 Decachlorobiphenyl			CAS #: 2051-24-3			
19.6	19.6	0.000	2221 0.00200	0.0026		(a)

8 Aroclor-1260			CAS #: 11096-82-5			
15.1	15.1	0.000	5765 0.10000	0.13	80.00- 120.00	100.00(a)

3/31/06

Data File: E2F1894F.D
Report Date: 31-Mar-2006 07:55

AMOUNTS						
RT	EXP RT	DLT RT	CAL-AMT	ON-COL	TARGET RANGE	RATIO
==	=====	=====	RESPONSE (ng)	(ng)	=====	=====
8 Aroclor-1260 (continued)						
16.4	16.4	0.000	9365 0.10000	0.12 151.74-	191.74	162.45
17.2	17.2	0.000	5721 0.10000	0.12 86.33-	126.33	99.24
Average of Peak Amounts =				0.12		

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1894R.D
 Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D
 Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
 Inj Date : 31-MAR-2006 02:31
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 18 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1660.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
5.01	5.01	0.000	1405 0.00200	0.0025		(a)

6	Aroclor-1016		CAS #: 12674-11-2			
5.92	5.91	0.010	1254 0.10000	0.12	80.00- 120.00	100.00(a)
8.25	8.24	0.010	1526 0.10000	0.12	112.38- 152.38	121.69
9.21	9.20	0.010	1023 0.10000	0.12	72.72- 112.72	81.58
Average of Peak Amounts =			0.12			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
17.0	17.0	0.000	2198 0.00200			(a)

8	Aroclor-1260		CAS #: 11096-82-5			
14.2	14.2	0.000	8966 0.10000	0.13	80.00- 120.00	100.00(a)

3/31/06

Data File: E2F1894R.D
Report Date: 31-Mar-2006 07:55

		AMOUNTS							
RT	EXP RT	DLT RT	RESPONSE (CAL-AMT	ON-COL	TARGET RANGE	RATIO		
==	=====	=====	ng)	(ng)	=====	=====		
8 Aroclor-1260 (continued)									
14.8	14.8	0.000	4745	0.10000	0.13	32.61-	72.61	52.92	
16.0	16.0	0.000	2397	0.10000	0.12	8.08-	48.08	26.73	
Average of Peak Amounts =					0.13				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Figure 2

Aroclor 1221 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D

Date : 30-MAR-2006 18:58

Client ID: AR1221FL3

Instrument: E2.i

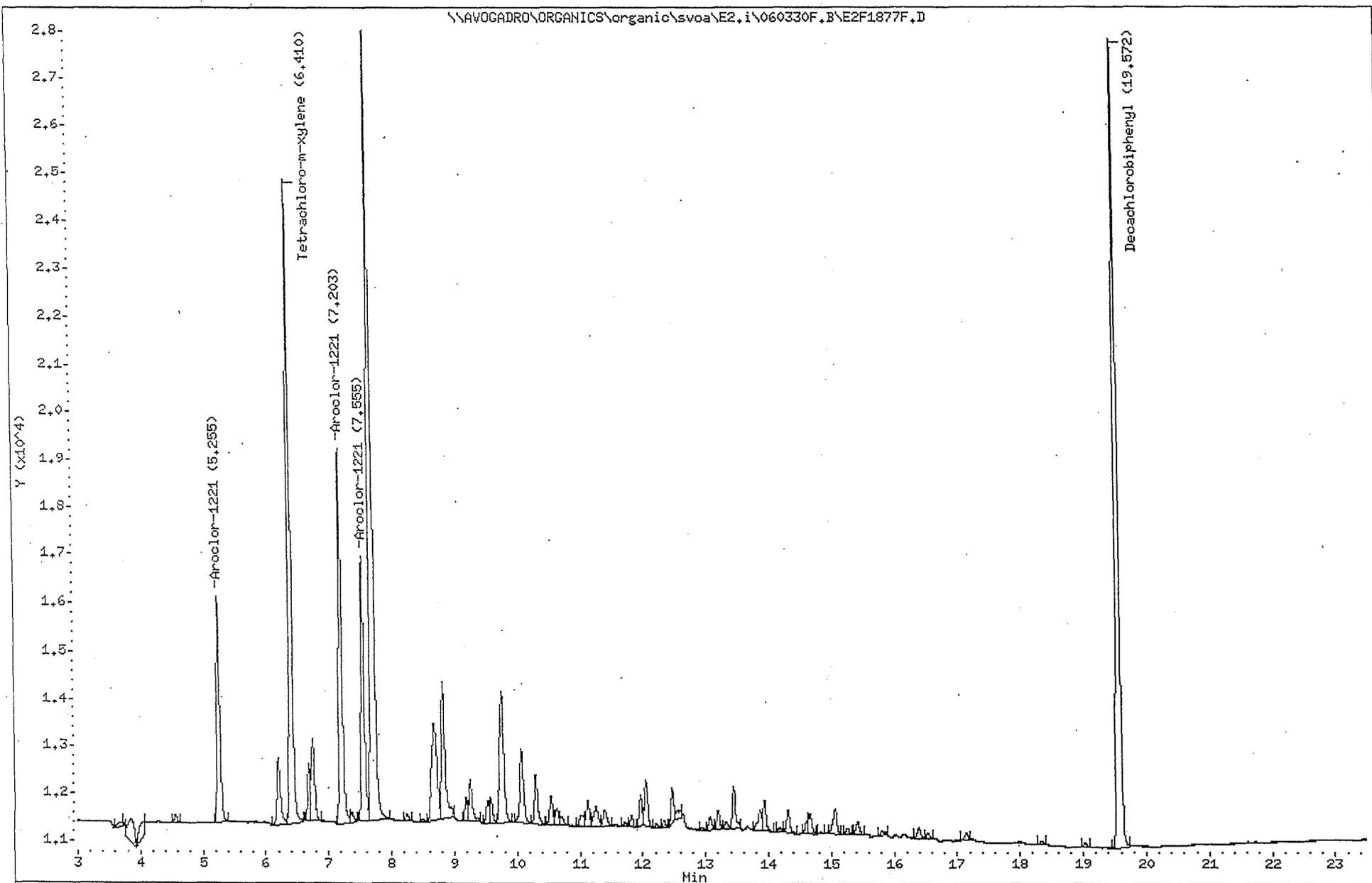
Sample Info: AR1221FL3,AR1221FL3,,ar1221,sub,,

Operator: SZ SRC: SZ

Volume Injected (uL): 1.0

Column diameter: 0.53

Column phase: RtxCLPPest



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2,i\060330R,B\E2F1877R.D

Date : 30-MAR-2006 18:58

Client ID: AR1221FL3

Sample Info: AR1221FL3,AR1221FL3,,ar1221,sub,,

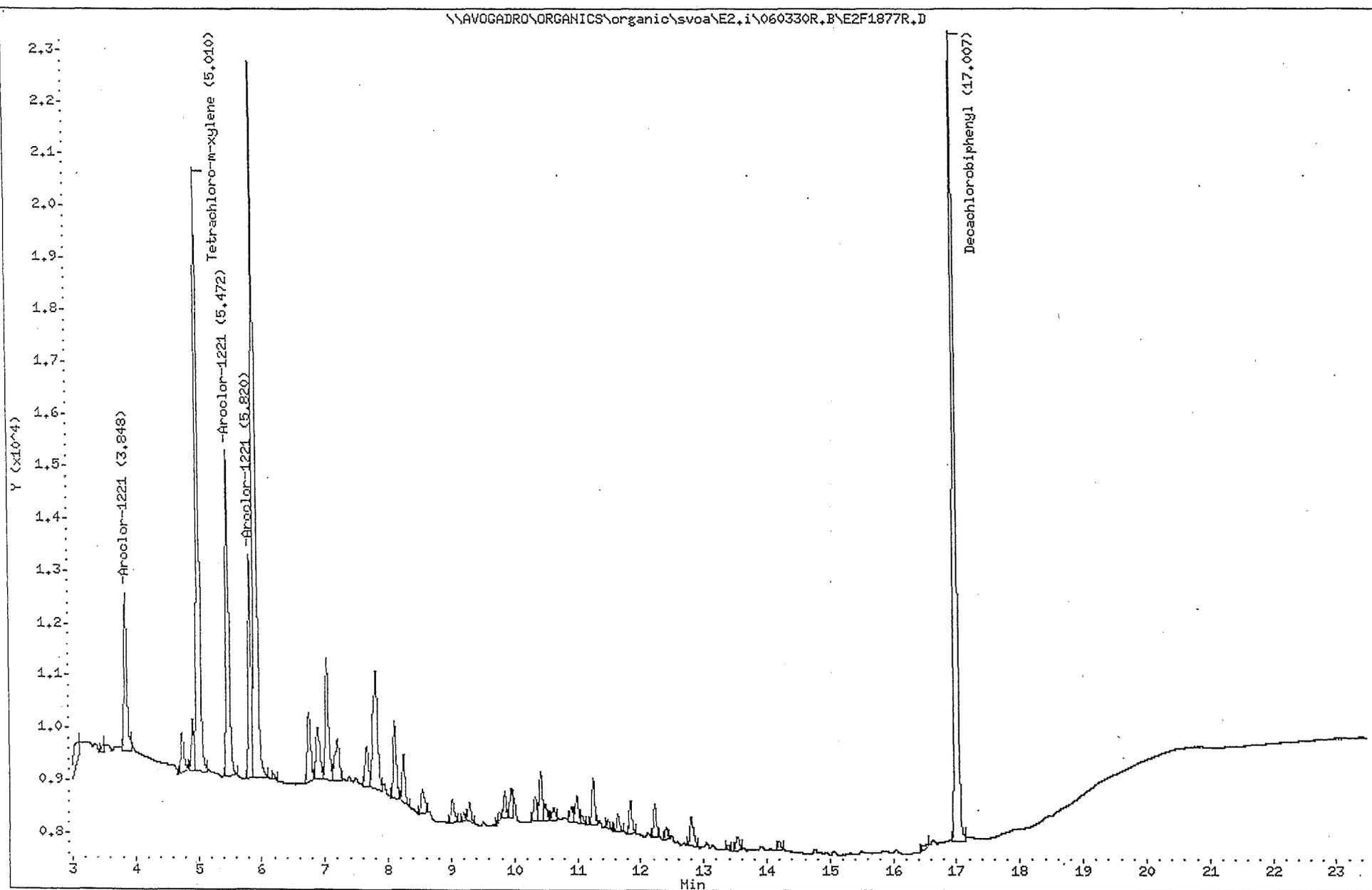
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1877F.D
 Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D
 Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
 Inj Date : 30-MAR-2006 18:58
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
 Misc Info : 1,3,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 1 Calibration Sample, Level: 3
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1221.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
6.41	6.41	0.000	13493 0.02000	0.020		(a)

2	Aroclor-1221		CAS #: 11104-28-2			
5.26	5.26	0.000	4734 1.00000	1.0	80.00- 120.00	100.00
7.20	7.20	0.000	7871 1.00000	1.0	146.27- 186.27	166.27
7.56	7.56	0.000	5525 1.00000	1.0	96.71- 136.71	116.71
Average of Peak Amounts =			1			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
19.6	19.6	0.000	16954 0.02000	0.020		(a)

3/31/06

Data File: E2F1877F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1877R.D
 Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1877R.D
 Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
 Inj Date : 30-MAR-2006 18:58
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
 Misc Info : 1,3,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 1 Calibration Sample, Level: 3
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1221.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
5.01	5.01	0.000	11534 0.02000	0.020		(a)

2	Aroclor-1221		CAS #: 11104-28-2			
3.85	3.85	0.000	3031 1.00000	1.0	80.00- 120.00	100.00
5.47	5.47	0.000	6227 1.00000	1.0	185.44- 225.44	205.44
5.82	5.82	0.000	4291 1.00000	1.0	121.57- 161.57	141.57
Average of Peak Amounts =			1			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
17.0	17.0	0.000	15520 0.02000	0.022		(a)

3/31/06

Data File: E2F1877R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Figure 3

Aroclor 1232 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D

Date : 30-MAR-2006 19:24

Client ID: AR1232FL3

Sample Info: AR1232FL3,AR1232FL3,,ar1232,sub,,

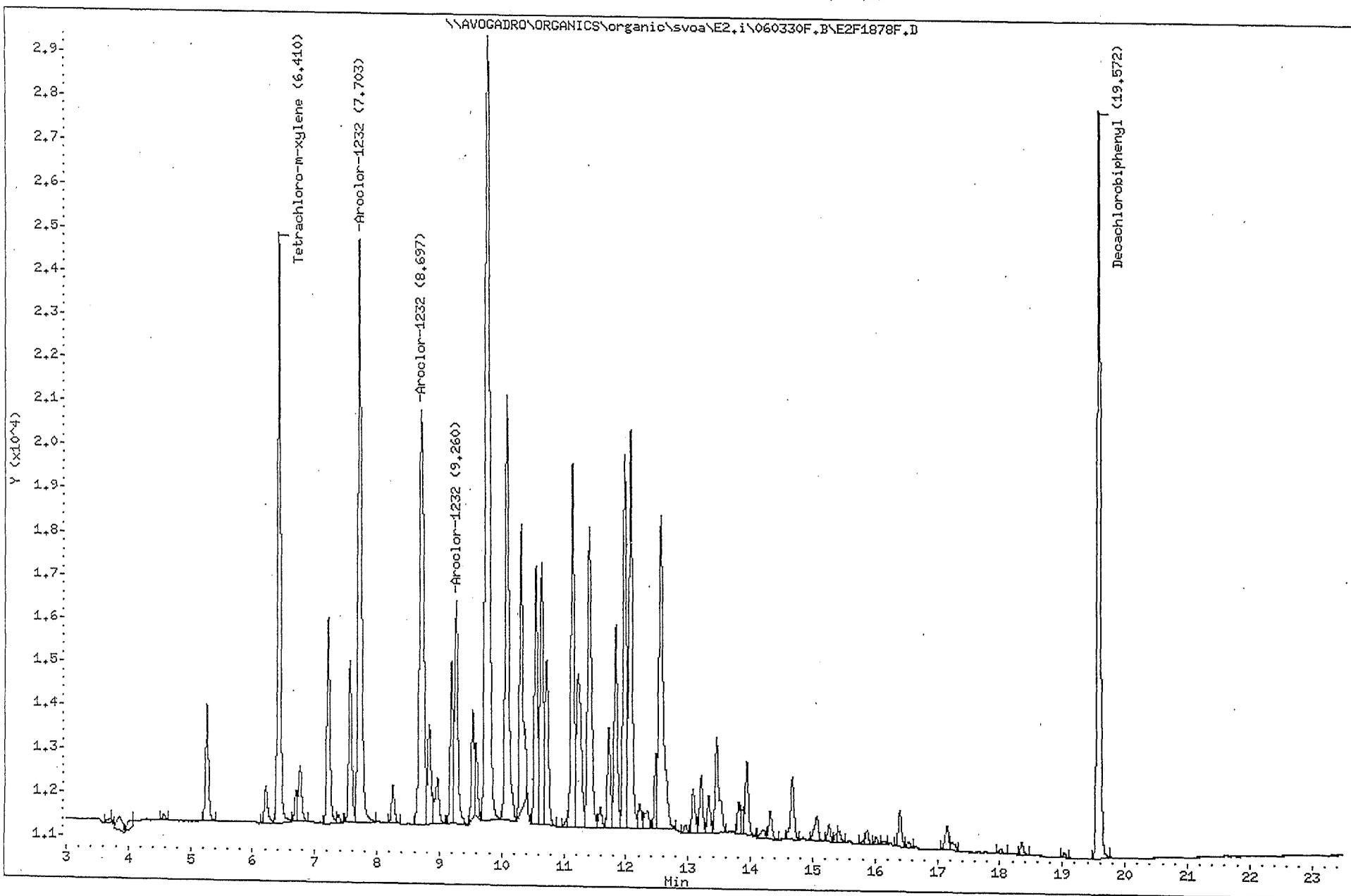
Volume Injected (uL): 1.0

Column phase: RtxCLPPest

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D

Date : 30-MAR-2006 19:24

Client ID: AR1232FL3

Sample Info: AR1232FL3,AR1232FL3,,ar1232.sub,,

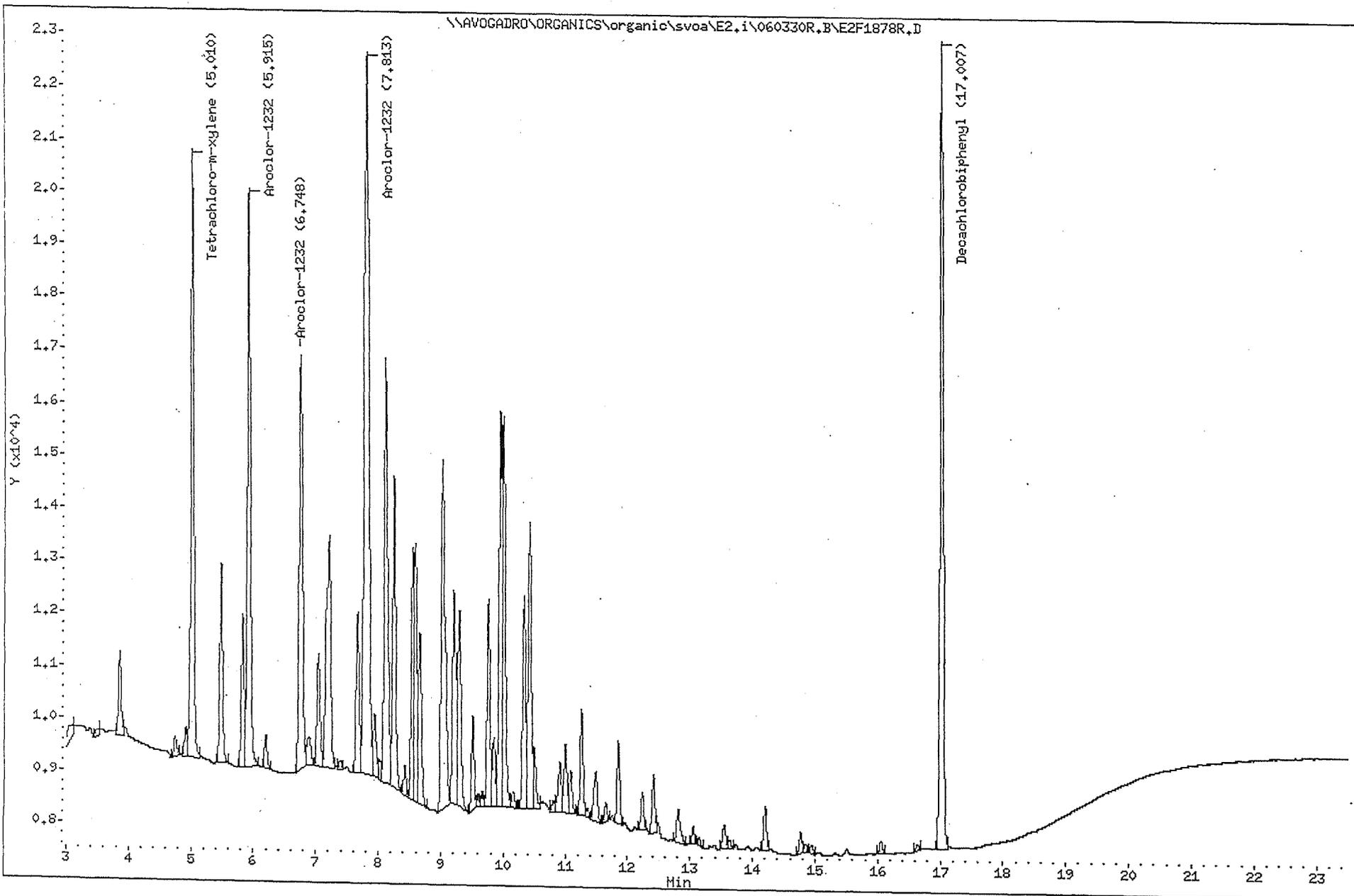
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1878F.D
 Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D
 Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
 Inj Date : 30-MAR-2006 19:24
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
 Misc Info : 1,3,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 2 Calibration Sample, Level: 3
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1232.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	RESPONSE (ng)	CAL-AMT (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene				CAS #: 877-09-8		
6.41	6.41	0.000	13570 0.02000	0.020			(a)

3	Aroclor-1232				CAS #: 11141-16-5		
7.70	7.70	0.000	13387 1.00000	1.0	80.00- 120.00	100.00	
8.70	8.70	0.000	9491 1.00000	1.0	50.90- 90.90	70.90	
9.26	9.26	0.000	5098 1.00000	1.0	18.08- 58.08	38.08	
Average of Peak Amounts =					1		

\$ 10	Decachlorobiphenyl				CAS #: 2051-24-3		
19.6	19.6	0.000	17061 0.02000	0.020			(a)

3/31/06 ✓

Data File: E2F1878F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1878R.D
 Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D
 Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
 Inj Date : 30-MAR-2006 19:24
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
 Misc Info : 1,3,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 2 Calibration Sample, Level: 3
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1232.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
5.01	5.01	0.000	11550	0.02000	0.020	(a)

3	Aroclor-1232		CAS #: 11141-16-5			
5.92	5.92	0.000	10974	1.00000	1.0 80.00- 120.00	100.00
6.75	6.75	0.000	7851	1.00000	1.0 51.54- 91.54	71.54
7.81	7.81	0.000	13684	1.00000	1.0 104.69- 144.69	124.69
Average of Peak Amounts =			1			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
17.0	17.0	0.000	15283	0.02000	0.022	(a)

3/31/06

Data File: E2F1878R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Figure 4

Aroclor 1242 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D

Date : 30-MAR-2006 19:51

Client ID: AR1242FL1

Sample Info: AR1242FL1,AR1242FL1,,ar1242,sub,,

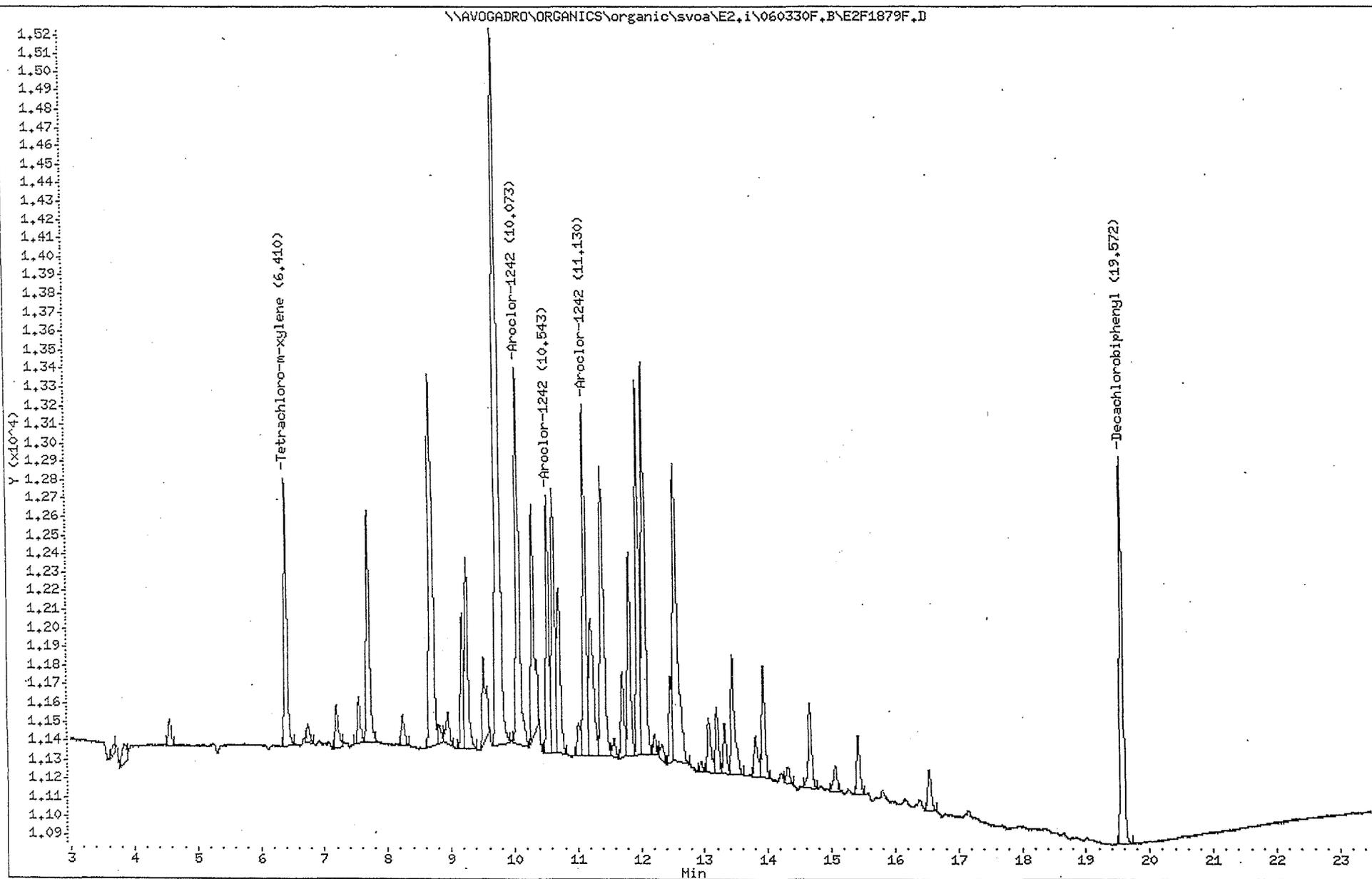
Volume Injected (uL): 1.0

Column phase: RtxCLPPest

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D

Date : 30-MAR-2006 19:51

Client ID: AR1242FL1

Sample Info: AR1242FL1,AR1242FL1,,ar1242.sub,,

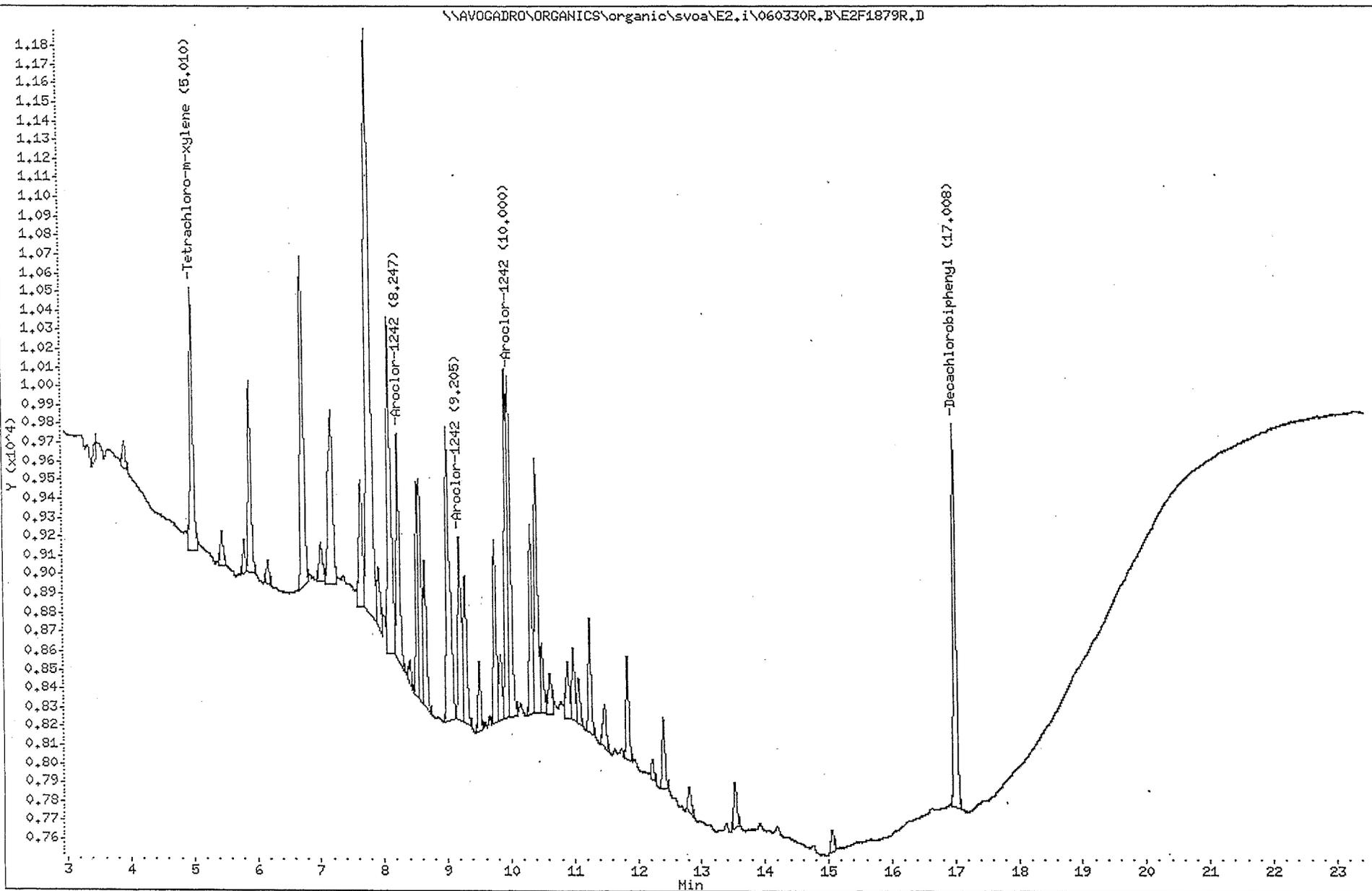
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1879F.D
 Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D
 Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
 Inj Date : 30-MAR-2006 19:51
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 3 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1242.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
6.41	6.41	0.000	1442 0.00200	0.0021		(a)

5	Aroclor-1242		CAS #: 53469-21-9			
10.1	10.1	0.000	2024 0.10000	0.12	80.00- 120.00	100.00 (a)
10.5	10.5	0.000	1380 0.10000	0.12	44.06- 84.06	68.18
11.1	11.1	0.000	1886 0.10000	0.12	71.10- 111.10	93.18
Average of Peak Amounts =			0.12			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
19.6	19.6	0.000	2073 0.00200	0.0024		(a)

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Data File: E2F1879F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1879R.D
 Report Date: 31-Mar-2006 07:52

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D
 Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
 Inj Date : 30-MAR-2006 19:51
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:52 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 3 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1242.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT	ON-COL	RESPONSE (ng) - (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8				
5.01	5.01	0.000	1392	0.00200	0.0024		(a)

4	Aroclor-1242		CAS #: 53469-21-9				
8.25	8.24	0.010	1190	0.10000	0.12	80.00- 120.00	100.00(a)
9.21	9.20	0.010	967	0.10000	0.12	62.62- 102.62	81.26
10.0	10.0	0.000	1808	0.10000	0.13	115.66- 155.66	151.93
Average of Peak Amounts =					0.12		

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3				
17.0	17.0	0.000	2030	0.00200			(a)

3/31/06

Data File: E2F1879R.D
Report Date: 31-Mar-2006 07:52

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Figure 5

Aroclor 1248 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F,B\E2F1884F.D

Date : 30-MAR-2006 22:04

Client ID: AR1248FL1

Sample Info: AR1248FL1,AR1248FL1,,ar1248,sub,,

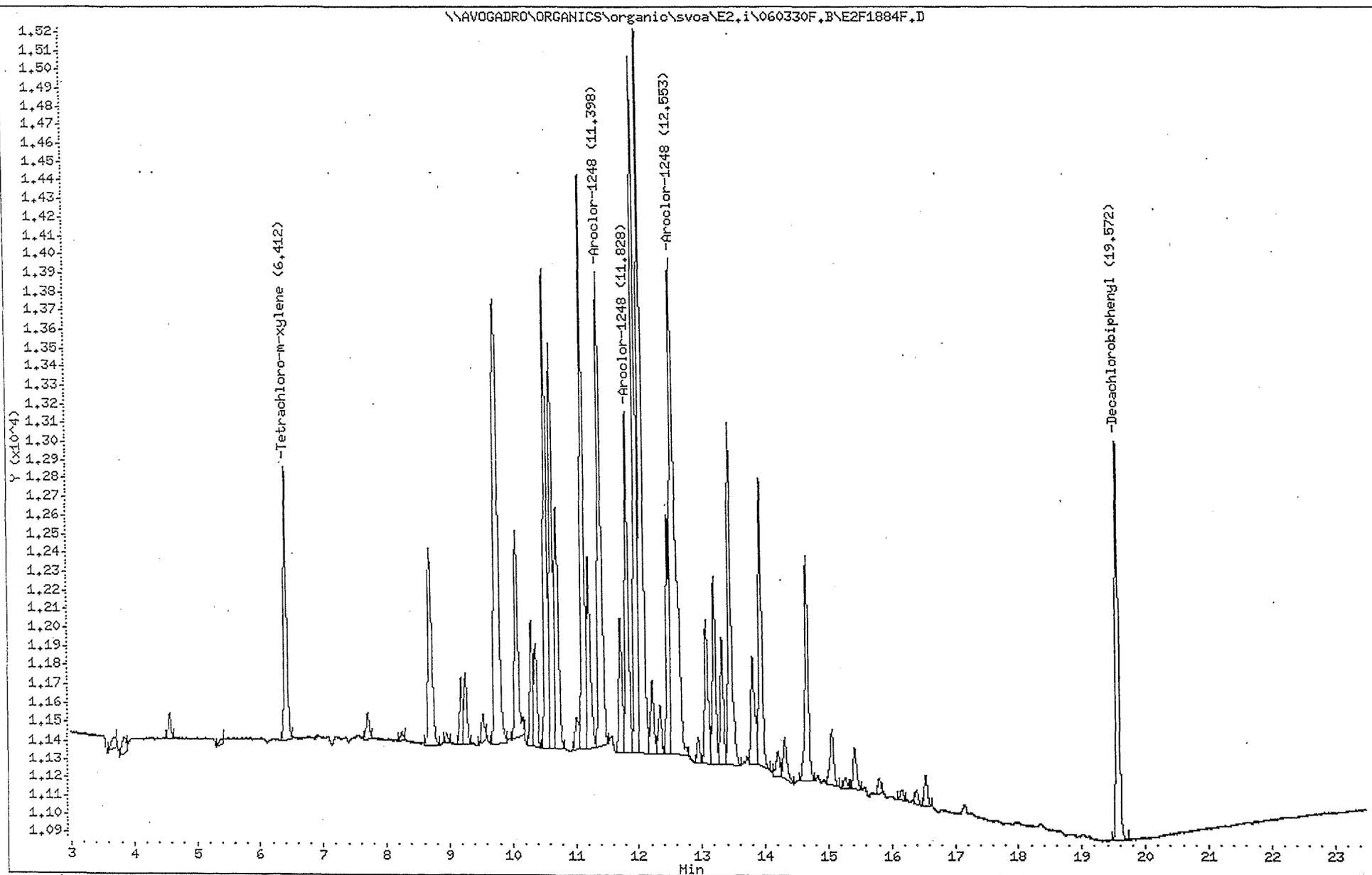
Volume Injected (uL): 1.0

Column phase: RtxCLPPest

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D

Date : 30-MAR-2006 22:04

Client ID: AR1248FL1

Sample Info: AR1248FL1,AR1248FL1,,ar1248.sub,,

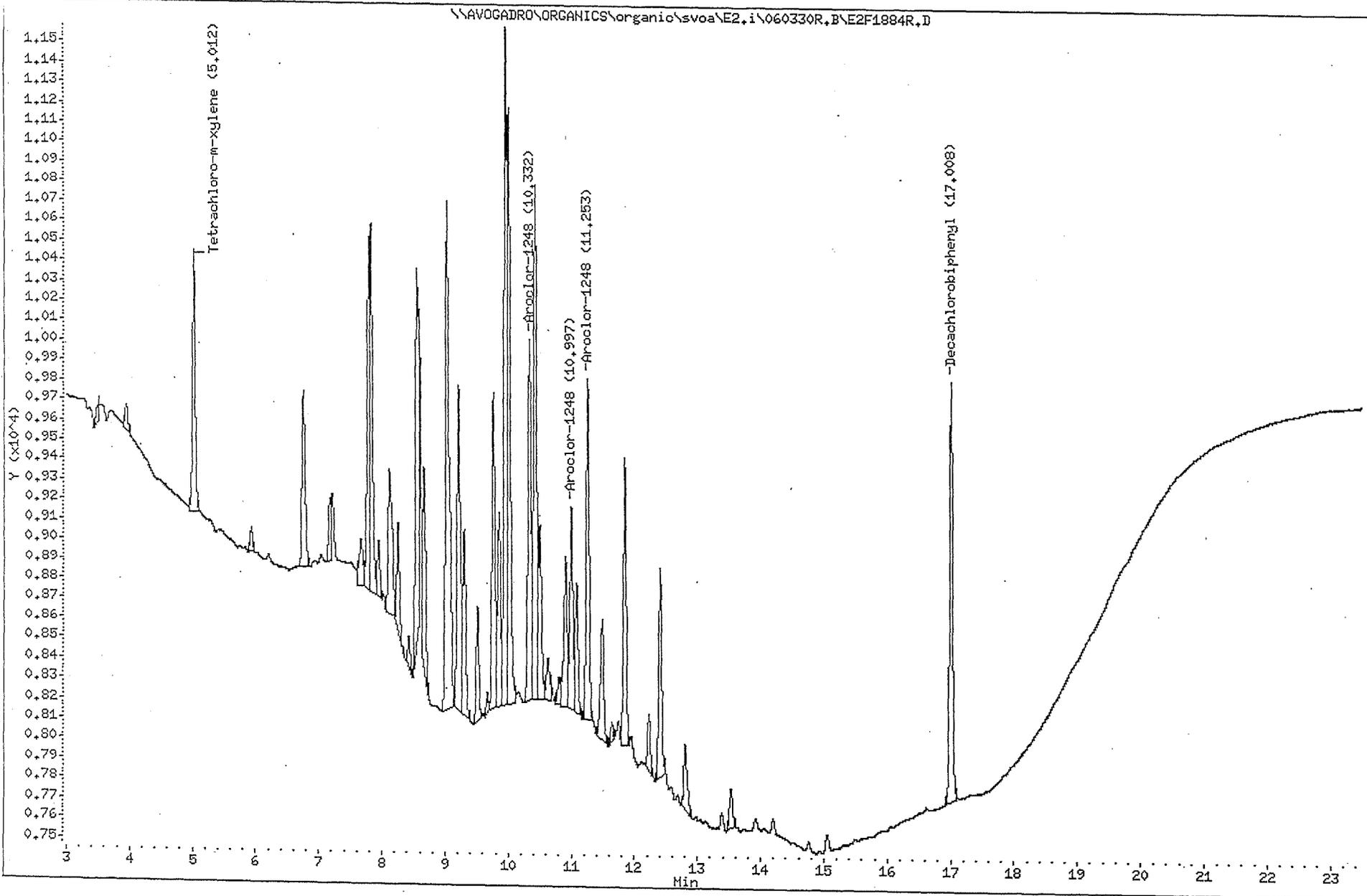
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1884F.D
 Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1884F.D
 Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
 Inj Date : 30-MAR-2006 22:04
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 8 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1248.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
6.41	6.41	0.000	1454 0.00200	0.0022		(a)

6	Aroclor-1248		CAS #: 12672-29-6			
11.4	11.4	0.000	2529 0.10000	0.12	80.00- 120.00	100.00(a)
11.8	11.8	0.000	1823 0.10000	0.12	50.48- 90.48	72.08
12.6	12.6	0.000	2646 0.10000	0.12	89.75- 129.75	104.63
Average of Peak Amounts =			0.12			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
19.6	19.6	0.000	2141 0.00200	0.0025		(a)

3/31/06 ✓

Data File: E2F1884F.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1884R.D
 Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D
 Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
 Inj Date : 30-MAR-2006 22:04
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
 Misc Info : 1,1,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 8 Calibration Sample, Level: 1
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1248.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$ 1	Tetrachloro-m-xylene		CAS #: 877-09-8			
5.01	5.01	0.000	1315 0.00200	0.0023		(a)

5	Aroclor-1248		CAS #: 12672-29-6			
10.3	10.3	0.000	1811 0.10000	0.13	80.00- 120.00	100.00 (a)
11.0	11.0	0.000	1016 0.10000	0.12	37.21- 77.21	56.10
11.3	11.3	0.000	1715 0.10000	0.13	72.29- 112.29	94.70
Average of Peak Amounts =			0.13			

\$ 10	Decachlorobiphenyl		CAS #: 2051-24-3			
17.0	17.0	0.000	2112 0.00200			(a)

3/31/06

Data File: E2F1884R.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Figure 6

Aroclor 1254 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D

Date : 31-MAR-2006 00:44

Client ID: AR1254FL2

Sample Info: AR1254FL2,AR1254FL2,,ar1254,sub,,

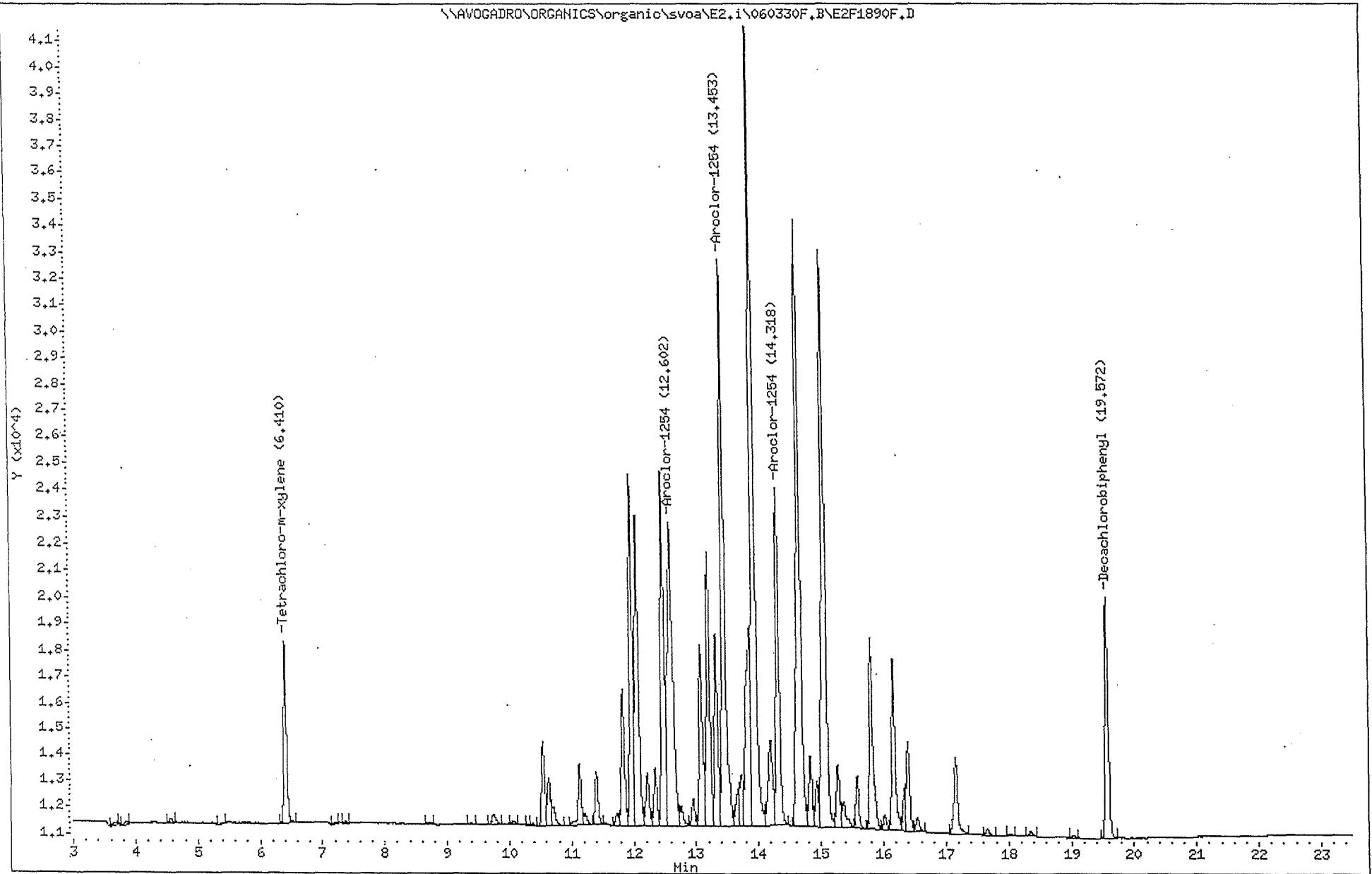
Volume Injected (uL): 1.0

Column phase: RtxCLPPest

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0,53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R,B\E2F1890R.D

Date : 31-MAR-2006 00:44

Client ID: AR1254FL2

Sample Info: AR1254FL2,AR1254FL2,,ar1254.sub,,

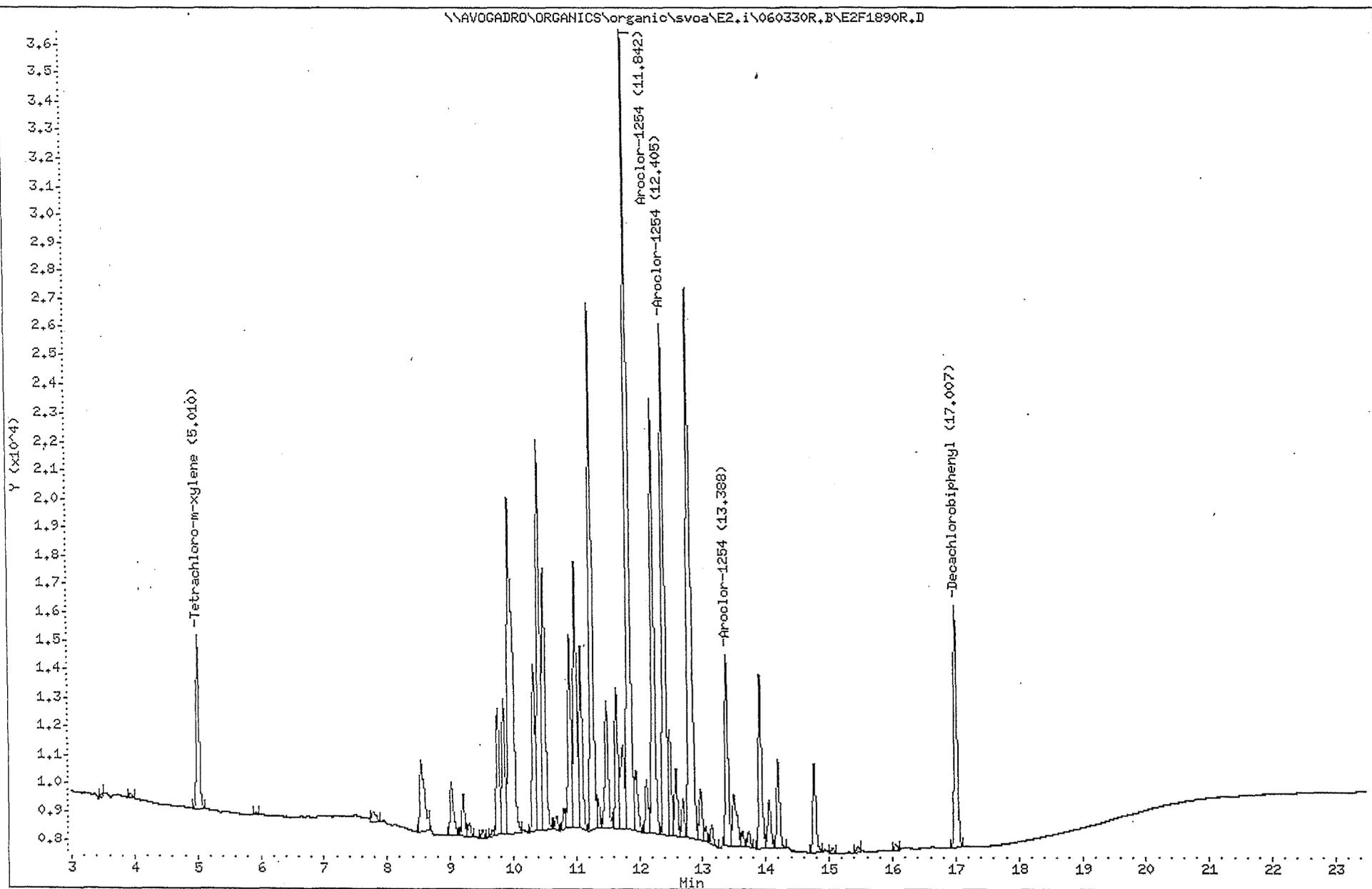
Volume Injected (uL): 1.0

Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ

Column diameter: 0.53



Data File: E2F1890F.D
 Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D
 Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
 Inj Date : 31-MAR-2006 00:44
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
 Misc Info : 1,2,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
 Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
 Als bottle: 14 Calibration Sample, Level: 2
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1254.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
UF	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	CAL-AMT RESPONSE (ng)	ON-COL (ng)	TARGET RANGE	RATIO
\$ 1					CAS #: 877-09-8	
6.41	6.41	0.000	6923 0.01000	0.010		(a)
\$ 10					CAS #: 2051-24-3	
19.6	19.6	0.000	9157 0.01000	0.011		(a)
7					CAS #: 11097-69-1	
12.6	12.6	0.000	11493 0.50000	0.54	80.00- 120.00	100.00(a)
13.5	13.5	0.000	21409 0.50000	0.54	171.37- 211.37	186.28
14.3	14.3	0.000	12764 0.50000	0.54	92.12- 132.12	111.06
Average of Peak Amounts =			0.54			

3/31/06

Data File: E2F1890F.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1890R.D
 Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1890R.D
 Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
 Inj Date : 31-MAR-2006 00:44
 Operator : SZ SRC: SZ Inst ID: E2.i
 Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
 Misc Info : 1,2,,1
 Comment :
 Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
 Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
 Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
 Als bottle: 14 Calibration Sample, Level: 2
 Dil Factor: 1.00000
 Integrator: Falcon Compound Sublist: ar1254.sub
 Target Version: 4.03 Sample Matrix: WATER
 Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt / (Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

AMOUNTS

RT	EXP RT	DLT RT	RT	RESPONSE (ng)	CAL-AMT (ng)	ON-COL (ng)	TARGET RANGE	RATIO

\$	1						CAS #: 877-09-8	
5.01	5.01	0.000		6063 0.01000	0.011			(a)

\$	10						CAS #: 2051-24-3	
17.0	17.0	0.000		8543 0.01000	0.010			(a)

	7						CAS #: 11097-69-1	
11.8	11.8	0.000		28134 0.50000	0.50	80.00-	120.00	100.00(a)
12.4	12.4	0.000		17965 0.50000	0.49	47.65-	87.65	63.86
13.4	13.4	0.000		6744 0.50000	0.50	5.17-	45.17	23.97
Average of Peak Amounts =				0.5				

3/31/06

Data File: E2F1890R.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Attachment 1

Corrective Action Examples and Documentation Tables

Attachment 1

Corrective Action and Documentation Examples

<u>OCCURRENCE</u>	<u>ACTION</u>	<u>DOCUMENTATION</u>
1. Initial calibration does not meet QC criteria ($RSD\% > 20\%$ or $r^2 < 0.990$).	1. Check GC conditions such as temperature program, makeup gas flow rate, and carrier gas flow rate. Check if column bleeding occurs and more injection maintenance is needed. After the reasons are found, appropriate maintenance will be done and a new initial calibration will be run. If the new curve still fails, the reasons for failing the curve will be re-evaluated and another a new curve will be rerun after the reevaluation. If still not good, call GC manufacturer.	1. Note in instrument run logbook, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification (ICV) check does not meet QC criteria ($D\% > \pm 20\%$).	2. Check the preparation of ICV standard, and if necessary, make a fresh ICV standard. If it is found that ICV standard is ok, the standards for initial calibration will be checked such as evaporation and the preparation of standards. A new ICV and a new curve will be analyzed after problems are corrected.	2. Note in instrument run logbook, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria ($D\% > \pm 20\%$).	3. Maintenance such as replacing septum and liner for injection port will be done, and then a new set of CCVs and the samples related to the bad CCVs will be re-analyzed. If the CCVs still fail, A full maintenance including replacing septum, liner and gold seal and trimming the column will be done and a new initial calibration will be run. Then the CCV and/or QC and samples re-analyzed.	3. Note in instrument run logbook. If instrument maintenance performed, note in maintenance logbook.
4. Method blank contains target compound above reporting limit.	4. A new aliquot of the blank from prep lab will be reanalyzed to check if contaminants are carried over from previous dirty samples and/or from standards or a bad injection occurs. If the results from re-analysis of the	4. If the re-extraction/re-analysis meets the criteria, the results are reported. Document in the run log. If there is insufficient sample volume for re-extraction/re-analysis, report the initial results. Document in the run log,

<p>5. Surrogates in the method blank are outside of acceptable range.</p> <p>6. Compound out of acceptance range in laboratory control sample.</p> <p>7. Surrogates in samples are out of control limits.</p>	<p>blank are still the same, whole batch of the samples that are related to the blank will be re-extracted and re-analyzed.</p> <p>5. A new aliquot of the blank from prep lab will be reanalyzed. If the results from re-analysis are the same, the samples that are related to this blank will be re-extracted and re-analyzed.</p> <p>6. Investigate source of problem. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP/project requirements, this LCS will be reanalyzed. If the results of reanalysis are the same, the samples that are related to the LCS will be re-extracted and re-analyzed.</p> <p>7. 7.1 No actions are needed for the following situations:</p> <ul style="list-style-type: none"> • One of the surrogates (TCX and DCB) is in control limits, and the other is above the upper limit due to coelution with contaminants. • TCX is in the control limits, and DCB is below the lower limit. This is possibly due to matrix effect. <p>7.2 Further actions are needed for the following cases:</p> <ul style="list-style-type: none"> • Both of the surrogates (TCX and DCB) do not meet criteria. • TCX is lower than the lower limits and DCB is 	<p>document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.</p> <p>5. Same as documentation #5.</p> <p>6. Flag all compounds out of range on Form 3 of data report, if samples are re-analyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook, comment in data review checklist to be included in project narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer. If re-analysis cannot be performed due to insufficient sample, comment in data review checklist to be included in project narrative, and document as above.</p> <p>7. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, comment on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. Document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer.</p>
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<p>10. Matrix spike recovery out of QC range.</p>	<p>10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in un-spiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, re-analyze spike sample.</p>	<p>10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p>
<p>11. Duplicate (or MSD) relative percent difference exceeds QC limit.</p>	<p>11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, re-analyze duplicate sample.</p>	<p>11. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p>
<p>12. Retention Time shift.</p>	<p>12. A. No further action is required if the retention times of the CCV and samples shift after regular maintenance such as replacing the septum, liner, gold seal and trimming of the columns. The retention times will be established.</p> <p>B. If the retention times of the CCV shift during analysis, GC maintenance will be performed and the CCV and associated samples and QC will be re-analyzed.</p> <p>C. If the retention times of the CCV do not shift, but the retention times of surrogates in some of the samples do shift, the samples will be re-analyzed. If the retention times still do not meet the criteria, matrix affect is assumed and the results will be reported. This situation will be documented.</p>	<p>12. Document in the run log. If CCV, QC, and samples were re-analyzed, document in the run log. If the retention times shift with respect to re-analysis, document in the run log, in the checklist for the case narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date.</p>

Attachment 2
Department of Defense QC Requirements

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	NA	NA	
Breakdown check (Endrin/DDT Method 8081 only)	Daily prior to analysis of samples	Degradation $\leq 15\%$ for both Endrin and DDT.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq 15\%$.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below (except for Method 8082, which may only use Option 1 or 2): Option 1: RSD for each analyte $\leq 20\%$ Option 2: linear least squares regression: $r \geq 0.995$ Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within $\pm 20\%$ of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (in Method 8081A exclude toxaphene and technical chlordane, in Method 8015B exclude GRO, DRO, and residual range organics (RRO)).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA	Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

TABLE D-15. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846
METHOD 8081 SOLID MATRIX²⁶

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
4,4'-DDD	81	18	30	135	10	155
4,4'-DDE	97	10	70	125	60	135
4,4'-DDT	92	16	45	140	30	155
Aldrin	93	16	45	140	30	155
alpha-BHC	93	10	60	125	50	135
alpha-Chlordane	92	10	65	120	55	130
beta-BHC	95	11	60	125	50	135
delta-BHC	94	12	55	130	45	145
Dieldrin	96	10	65	125	55	135
Endosulfan I	74	20	15	135	10	155
Endosulfan II	89	17	35	140	20	160
Endosulfan sulfate	99	12	60	135	50	145
Endrin	97	12	60	135	50	145
Endrin aldehyde	92	18	35	145	20	165
Endrin ketone	100	11	65	135	55	145
gamma-BHC	91	11	60	125	50	135
gamma-Chlordane	96	10	65	125	55	135
Heptachlor	96	15	50	140	35	155
Heptachlor epoxide	98	11	65	130	55	140
Methoxychlor	100	14	55	145	45	155

TABLE D-16. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846
METHOD 8082 WATER MATRIX²⁷

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	85	20	25	145
Aroclor 1260	87	19	30	145

TABLE D-17. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846
METHOD 8082 SOLID MATRIX²⁷

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	90	16	40	140
Aroclor 1260	96	12	60	130

²⁶ A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

²⁷ LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

For

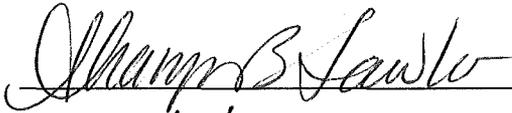
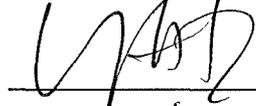
Gasoline Range Organics in Aqueous and Soil Samples

By

Gas Chromatography-Flame Ionization Detector (GC-FID)

using Methods SW-846 8015 and Maine HETL 4.2.17

Rev. 11

	Signature	Date
QA Director:	 _____	<u>6/8/10</u>
Lab Director:	 _____	<u>6/9/10</u>
Effective Date:	<u>6/16/10</u>	

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

For

**Gasoline Range Organics in Aqueous and Soil Samples by
Gas Chromatography-Flame Ionization Detector (GC-FID)
using Methods SW-846 8015 and Maine HETL 4.2.17**

Rev. 11

1. Scope and Application

This Standard Operating Procedure (SOP) describes the analysis of gasoline range petroleum hydrocarbon products in aqueous and soil samples using Gas Chromatography with a Flame Ionization Detector (GC-FID) according to protocols discussed in SW846 Method 8015 and Maine Method HETL 4.2.17. These methods are used in conjunction with Purge and Trap sample introduction technique. The Gasoline Range Organics (GRO) methods facilitate the general identification and quantitative analysis of hydrocarbons derived from petroleum distillate products with boiling points approximately equal to or less than that of gasoline.

This SOP is developed to follow the guidelines specified in the SW846 methods. To further familiarize with the procedures, the analyst is highly encouraged to consult the following instrument manuals as well as the methods themselves (see reference section):

- Hewlett Packard HP 5980 Gas Chromatograph with Flame Ionization Detector Manual
- Hewlett Packard EnviroQuant Manual
- OI Corporation Manuals on Model 4560 Purge and Trap Concentrator and Model 4551A Autosampler
- OI Corporation Manual on Model 4410 Flame Ionization Detector

2. Personnel Qualifications and Responsibilities

Laboratory analysts must be sufficiently trained and familiarized with the GC instrumentation and software in order to perform sample testing and analysis, as well as instrument maintenance and troubleshooting, to a degree considered sufficient to accomplish the tasks necessary with a reliable level of accuracy and efficiency.

The analysts may revise the SOP in conjunction with the QA Director as necessary to improve upon the Gasoline Range Organics method or to accommodate for any changing conditions that would affect the performance of the method.

3. Summary of Procedure

Samples containing volatile compounds are loaded onto a Model 4551A autosampler and are introduced into the GC by purge-and-trap system. The analytes are desorbed onto the column directly. The column is temperature programmed to separate the compounds, and they are then detected using a flame ionization detector.

The GRO compounds' responses are compared to the collective response of a six-point calibration curve in order to quantify the concentration of gasoline range organics in the sample being tested.

4. Sample Preservation, Containers, Handling and Storage

4.1 Water samples are collected in the field in 40mL glass vials with a Teflon septum. Two glass vials for each water sample should be provided in order to accommodate re-analysis. The water sample is preserved in the field to a pH of <2 with HCl. When received, samples are refrigerated to a temperature of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and are stored in the lab at that same temperature.

4.2 Soil samples are generally collected into pre-weighed VOA vials containing 5mL of purge and trap grade methanol. The vials are re-weighed at the laboratory to determine the sample weight. Mitkem typically purchases pre-weighed vials from the company's bottle supplier. These are available in either 5mL or 10mL methanol aliquots, as printed on the bottle label. The weight of sample is not critical, but the ratio of sample to methanol should be 1:1, and the soil should be completely covered by methanol. The client is instructed not to add additional labels to the sample vial. A separate aliquot of unpreserved soil needs to be collected for the determination of percent moisture, as described in a separate SOP. In areas where soil samples for VOC analysis are not required to be preserved (New York State as of January, 2006 for example), samples may also be collected in 2 or 4 ounce glass containers and are stored at $4^{\circ}\text{C} \pm 2$.

4.3 The samples are recorded in the laboratory-tracking log upon receipt.

4.4 All samples must be analyzed within 14 days of collection.

5. Interferences and Potential Problems

Interference can occur with non-gasoline components eluting within the GRO range, such as certain chlorinated and oxygenated hydrocarbons. If it is known or suspected that non-gasoline interferences are present, the analyst should suggest additional analysis.

Sample contamination can occur when volatile organic compounds diffuse through the septum of the sample container during transport or storage. Trip blanks may be provided for analysis if such contamination is expected to occur.

Contamination may also occur due to carryover of compounds from a high concentration sample to a low concentration sample if sequentially analyzed. Care should be taken with the cleaning procedures of syringes and other sample handling equipment in order to be sure carryover does not occur. Carryover may also occur if a high concentration sample contaminates the sample sparger and trap. Screening of samples is recommended to protect laboratory equipment and to prevent carryover. If carryover occurs or is suspected, a re-analysis of the affected sample must be done. Proper cleaning and baking of the trap and sample sparger should be done after a concentrated sample in order to prevent further carryover contamination from occurring.

6. Equipment and Apparatus

A Hewlett Packard Model 5890 GC fitted with a Flame Ionization Detector (FID) or an OI Model 4410 Flame Ionization Detector in series with an OI Model 4430 Photoionization Detector (PID) is used for the analysis. The GC is connected to an OI Model 4560 Purge and Trap Concentrator and an OI Model 4551A Autosampler. With the PID-FID detectors in series, the PID is not used during GRO analysis. Compounds of interest are swept through the PID into the FID for analysis.

The GC operates under the control of a Hewlett Packard Chemstation System consisting of a Hewlett Packard Vectra PC. Hewlett Packard EnviroQuant Software is used to control both data acquisition and data reduction.

The following accessories are used with the GC system:

a) Column: DB-624, 105m x 0.45 mm ID, 2.55um film, Cat.124-1374, J&W Scientific.

b) The GC-FID operating conditions are as follows:

Column linear velocity	30cm/min
Make-up gas flow rate	90mL/min
Injector Temp	N/A
Detector	250°C
Initial GC Temp	40°C
Initial GC Time	1.0 min
Initial Temp Ramp	10°C/min

Secondary GC Temp	160°C
Secondary GC Hold	2.0 min
Secondary Temp Ramp	20°C/min
Final GC Temp	240°C
Final GC Hold	8.0 min

c) Purge and Trap Conditions:

Trap Material	Tanax/silica
Purge Gas Flow Rate	40mL/min
Purge Temp	35°C
Purge Time	11 min
Dry Purge	0 min
Desorb Preheat	100°C
Desorb Temp	190°C
Desorb Time	2 min
Bake Temp	210°C
Bake Time	15 min

Maintenance schedules and procedures for all of the equipment used in the GRO method should be followed as described in each piece of equipment's respective manual. All non-routine maintenance performed should be documented in the instrument maintenance logbook, located in LIMS. Routine maintenance should be documented in the instrument run log.

7. Reagents and Standards

The following description is based on stock standards purchased from the quoted sources. Equivalent standards from other commercial sources can be used as long as the standards are traceable to sources that can be compared to reference materials. It is also important that only high purity standards (> 96% purity) be used.

- 7.1 Surrogate Standard:
p-Bromofluorobenzene, Restek (Cat. No. 30003) at 5,000µg/mL.
- 7.2 GRO Standard:
PVOC/GRO Mix (Wisconsin), Ultra (Cat. No. UST-100) at 1000µg/mL.
- 7.3 GRO Laboratory Control Sample (LCS)/ Initial Calibration Verification(ICV):
Unleaded gasoline composite standard, Restek (Cat. No.30081) at 2,500µg/mL.
- 7.4 Analyte-Free Reagent Water (ASTM Type II water) – prepared by filtering tap water through a column of activated charcoal granules.

7.5 Purge and trap grade methanol, Fisher Scientific or another source of equivalent quality, is used for standard preparation.

7.6 Gasses used for the instrumentation and detectors are listed below:

Carrier Gas and Purge Gas	99.999% Helium
FID gas	Hydrogen (0.5 Grade) at about 25mL/min. Air at about 100mL/min.

8. Procedure

8.1 Standard preparation: All standard preparations are documented in the Primary, Intermediate and the Working Standard Logbooks located in the lab or in LIMS.

8.1.1 Primary Standards:

All primary standards received from vendors are logged into the Volatiles Primary Standard Logbook. The standards are labeled PVyyymmddX where:

PV = Primary Volatile standard,

yyymmdd = date the standard is received, and

X = the order the standard is logged into the Logbook on that date, in increasing alphabetical order.

Thus, VP990725C represents the third primary Volatile standard logged into the Primary Volatile Standard Logbook on July 25, 1999.

Where applicable, certificates of analysis which accompany the standards should also be labeled with the same identifiers and archived together to facilitate tracing of the standards. Primary standards or stock standards are good for up to 6 months if stored with minimal headspace under refrigeration. Replace sooner if comparison to check standards indicates an issue.

8.1.2 Working Standards:

All of the working standards are logged into the Volatiles Working Standard Logbook and labeled as VWyyymmddX where:

VW = Volatile Working standard

yyymmdd = date the working standard is prepared, i.e., 070719 for a standard prepared on July 19, 2007

X = the order that the working standard is prepared on that date, in increasing alphabetical order

The working standards are stored in amber vials fitted with Teflon septa. They are stored in the freezer (-10°C to -20°C) to minimize volatilization losses. The standards are replaced every 30 days or sooner if degradation is suspected.

A smaller or larger volume of the primary stock solution may be used to prepare the surrogate standard working solution. The final volume will be adjusted accordingly to achieve the same working concentration.

8.1.2.1 Surrogate Standard (BFB):

The working surrogate standard is prepared by combining 80µL of the primary p-Bromofluorobenzene standard at 5,000µg/mL and diluting to 4mL using methanol. Final concentration of the surrogate standard solution is 100µg/ml. The addition of 1µL of this solution to 5mL sample aliquot gives a surrogate concentration of 20µg/L on column.

8.1.2.2 GRO working standard:

The GRO working standard is prepared by taking 100uL of the primary standard at 1000µg/mL and diluting it to 1ml with methanol. The final concentration of the working GRO standard is 100µg/mL. The addition of 20µL of this solution to a 40mL aliquot gives a Standard concentration of each individual component is at 50µg/L on column, and GRO at 500µg/L on column.

8.1.2.3 GRO ICV (Gasoline Composite Standard):

The instrument is calibrated using a mixture of gasoline components (individual compounds). The initial calibration is verified using a gasoline product standard. The LCS and MS/MSD are also typically prepared using this gasoline product standard. The GRO ICV is prepared by taking 80µL of the primary standard at 2500µg/mL and diluting it to 1.0 mL with methanol. The final concentration of the working GRO ICV is 200µg/mL. The addition of 100µL of this solution to a 40mL aliquot gives a standard concentration of 500µg/L on column.

8.2 Initial Calibration:

The six levels of the calibration curve are loaded onto the autosampler and purged. The six levels used are at concentrations of 2.5, 5, 20, 50, 100, and 200µg/L per component.

Preparation is as follows:

<u>Standard Conc. (µg/L)</u>	<u>Amount Working Std. Used (µL)</u>	<u>BFB(µL)</u>
2.5	1	1
5.0	2	1
20	8	1
50	20	1
100	40	1
200	80	1

The concentrations above are diluted to 40mL using analyte-free reagent water. A typical chromatogram (10 components at 50µg/L) is shown in **Attachment 1** with the Integration Report.

8.2.1 Procedure for Initial Calibration:

Purge each calibration standard using the same technique that will be applied to the samples. Tabulate the peak height or peak area responses against the concentration of each individual ten compounds and BFB.

8.2.1.1 The response Factor (RF) is determined as follows:

$$RF = \frac{A_x}{C_x}$$

where: A_x = peak area of BFB or gasoline component to be measured

C_x = concentration of BFB or gasoline component to be measured

8.2.1.2 If the RF value over the working range is constant ($\leq 20\%$ RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations.

8.2.1.3 The % Relative Standard Deviation (%RSD) of the RF is also calculated using:

$$\% RSD = \frac{\text{Standard Deviation}}{\text{RF}} \times 100$$

Mean

where: Standard Deviation = $\sqrt{\sum (X_i - X)^2 / (n-1)}$

where: X_i = each individual value used to calculate the mean

X = the mean of n values

n = the total number of values

8.2.1.4 A collective response factor must also be established for the entire hydrocarbon range. To calculate the collective RF, tabulate the summation of the peak areas of all 10 components against the total mass injected (do not include the BFB peak area). The collective RF is the average of the responses for the Gasoline Range Organics (GRO) individual components across the entire range.

8.2.1.5 The Initial Calibration also sets the retention times necessary for the (GRO) integration for field and QC samples. The RT_i is set at the time where methyl-tertiary-butyl ether (MTBE) starts to elute, and RT_f is set after the elution of naphthalene. RT_i and RT_f are saved as reference points and will be used for all subsequent instrument and method blanks, lab control samples, duplicate matrix spikes and sample analysis.

8.2.1.6 The area count of BFB is independently integrated.

8.2.2 Each initial calibration is verified by the analysis of a standard from an independent second source. This is typically a gasoline product standard. This initial calibration verification (ICV) must be within +/- 20% of the true value for the initial calibration to be acceptable.

The ICV integration is performed by constructing a baseline from the point (RT_i) on the chromatogram where the gasoline components start to elute (methyl-tertiary-butyl ether (MTBE)) to a point after the hydrocarbons have completely eluted off the GC column (naphthalene) (RT_f). The area of the surrogate, BFB, is subtracted from the total GRO area prior to calculating the GRO concentration.

A gasoline product standard chromatogram and integration report are shown in **Attachment 2**.

8.3 Continuing Calibration:

Continuing Calibration standards are analyzed to ensure that the GC-FID continues to meet instrument sensitivity and linearity requirements.

8.3.1 Frequency of Continuing Calibration:

The Continuing Calibration standard must be performed once every 12 hours, or after 20 injections, whichever is more frequent. If time remains in the 12 hour time period after meeting the acceptance criteria for the Initial Calibration, samples may be analyzed. It is not necessary to analyze continuing calibration standards if the Initial Calibration standards meet the Continuing Calibration acceptance criteria. A continuing calibration standard is also required at the end of the analytical sequence.

8.3.2 Procedure for performing Continuing Calibration:

The Continuing Calibration is analyzed at 50µg/L.

Calculate the percent difference between the Continuing Calibration RF and the values from the most recent initial Calibration.

The percent difference is determined as follows:

$$\% \text{ Difference} = \frac{\text{RF}_c - \text{RF}_i}{\text{RF}_i} \times 100$$

where: RF_c = response factor from Continuing Calibration

RF_i = mean relative response factor from the most recent Initial Calibration

8.4 Analytical Sequence:

The order used for the testing of samples is as follows:

1. Initial Calibration if necessary or Continuing Calibration
2. Instrument/Method Blank
3. Laboratory Control Sample/Duplicate
4. Sample
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
18. Sample
19. Sample
20. MS
21. MSD
22. Ending CC, etc.

This sequence is documented in the Volatiles Instrument Run Log (**Attachment 3**).

8.5 Initial Procedure:

8.5.1 Hardware Start-up:

The Gas Chromatograph, Sample Concentrator, and Multi-sampler should all be turned on, and the Gas Chromatograph and Sample concentrator should be allowed to reach their proper operating equilibrium temperatures.

The method file in the Sample Concentrator which is appropriate for the GRO method should be loaded from memory, and the detectors for the Gas Chromatograph should be on and operational. Make sure the Flame Ionization Detector has been lit.

8.5.2 Software Start-up:

On the computer that controls the Gas Chromatograph and Sample Concentrator, run the appropriate Enviroquant programs that are needed for sample running and processing.

Make sure a sequence has been entered properly in the Enviroquant software that will give all information on sample identification and concentration, and is in the same order in which the samples are to be run.

The Sample Concentrator needs to have the appropriate sequence entered into memory that will match the sequence entered into the Enviroquant software.

8.5.3 Sample loading:

8.5.3.1 Instrument/Method Blanks: Aqueous Method Blanks are prepared by spiking 1 μ L of the BFB spiking solution into 5mL of analyte-

free water. Soil Method Blanks are prepared by spiking 1 μ L of the BFB spiking solution and 100 μ L of methanol into 4.9mL of analyte-free water. An Instrument Blank may be used when appropriate to subtract out baseline interferences from samples associated with the instrument. An instrument Blank is prepared exactly the same as the Method Blank, according to matrix. A method blank can double as an instrument blank if needed. The Blanks are analyzed using the same procedure as that for sample analysis.

8.5.3.2 Aqueous samples: Samples are analyzed in 5mL aliquots. The 40ml vial is placed in a location in the autosampler tray. Immediately prior to analysis, an aliquot of the sample is withdrawn from the VOA vial by the autosampler using a syringe. An aliquot of the surrogate is added and the sample aliquot is transferred to the purge-and-trap sparger and injected into the sparger vessel. The sample is ready for analysis.

8.5.3.3 Soil samples: Mix the soil/methanol preserved aliquot by shaking and allow to settle. Note in the preparation logbook if the soil is not covered by the methanol layer.

Weigh the vial, being sure to tare-out the weight of the Mitkem sample label. Record the weight in the Soil Weight Logbook. Also record the initial tare weight of the preservative/vial printed on the sample label by the bottle supplier.

Open the vial and remove 800 μ L into a 40ml VOA vial with VOA-free water and then follow the same procedure of aqueous samples.

8.5.3.4 For unpreserved soils, weigh 5.0 to 5.5g of soil sample into a pre-weighed VOA vial containing 5mL of methanol. For Maine GRO samples received as unpreserved soils, the method requires a minimum of 10 grams of soil to be extracted, with a 1:1 ratio of soil to methanol. Shake vigorously and allow contents to settle. Analyze per procedure above.

8.5.4 Sample Analysis:

Once enough of the samples are loaded and the sequence is entered properly, start the sequence in the Enviroquant software and on the autosampler. The samples are analyzed using the previously listed GC conditions in the Equipment and Apparatus section.

Note: While both may be started before all the samples are loaded, make sure all necessary samples are loaded in the proper positions on the autosampler.

8.5.5 Quantitation of Samples:

From the chromatogram, the area of the peaks is integrated using the RT_i to RT_f approach. The sample integration is performed by constructing a baseline from a point (RT_i) on the chromatogram where the gasoline components start to elute to a point after the hydrocarbons have completely eluted off the GC column (RT_f). RT_i and RT_f are established from the initial calibration, and used for all method blanks, lab control samples, duplicate matrix spikes and sample analysis. For samples that contain higher boiling distillates such as diesel, only those hydrocarbon components within RT_i and RT_f are included for the quantitation.

8.5.6 Data Processing:

8.5.6.1 Once a run is complete, the data file will be stored in the computer. Load the appropriate file, and process the sample using the Enviroquant software. If the sample is a calibration level, update the five point calibration curve or the continuing calibration as necessary before processing any other samples.

8.5.6.2 After processing, the GRO are manually integrated using the Enviroquant software, as described in **section 8.5.5**. This is because the GRO compounds cover a range that is too extensive to be integrated by the Enviroquant software itself. After the GRO compounds are properly integrated, check the integration of the surrogate standard to be sure it is also integrated properly. The area of the surrogate will be subtracted from the area of the entire GRO range for sample concentration calculations. The analyst should feel free to perform manual integrations as necessary, but only if such integrations follow the guidelines put forth in this document.

8.5.6.3 When necessary, the Instrument/Method Blank area is subtracted from ALL field and QC sample analyses run in the associated sequence by the Enviroquant software. The area of integration from the instrument blank is entered in N4 under user defined items. Open the GC data acquisition software, under Initial Cal> Edit compounds> View GRO> Page 2. After the entire sequence has been processed, type MAC "TPHBS" at the bottom of the data acquisition window.. Then type DOLIST "THPBS". Select the files you want to subtract the IBLK from and process.

8.5.7 Analysis of Data:

After the sample is properly processed and integrated, the analyst must check to see if the sample passes the acceptance criteria appropriate for that particular type of sample. The results of the analysis are then recorded in the instrument run log, as well as any corrective action necessary (i.e. OK, needs dilution...). Corrective action responses are detailed in **section 12**.

9. Data Reduction and Calculations

9.1 Integration Range:

The concentration of Gasoline Range Organics is determined from a summation of the total peak area for all chromatographic peaks eluting from methyl-tertiary-butyl-ether (RT_D) to naphthalene (RT_F), inclusive less the area of the surrogate standard. Quantitation is based on a collective average response from the calibration curve.

9.2 Calculation:

Qualitative identification of the petroleum product, if requested by the client, is made by comparing the sample chromatogram to those of the standard gasoline chromatograms.

Quantitate the sample as follows:

$$\text{Aqueous sample concentration (in mg/L)} = \frac{A_x}{RF_i} * DF$$

where: RF_i = Mean relative response factor from the most recent calibration.

A_x = Peak area of gasoline or BFB to be measured.

DF = Dilution Factor.

$$\text{Soil sample concentration (in mg/Kg)} = \frac{(A_x) (5) (\underline{AV}_i)}{(RF_i) (\underline{V}_a) (W_s) (\%S)} * DF$$

where: RF_i = Mean relative response factor from the most recent calibration.

A_x = Peak area of gasoline or BFB to be measured.

W_s = Volume of sample used for analysis, in g.

%S = percent solids as calculated below.

V_a = volume of the aliquot of the sample methanol extract, in mL

V_t = total volume of methanol extract, in mL

AVt = Adjusted total volume of the methanol extract plus soil water in mL

determined by : $AVt = Vt + W_s * D$

D = percent moisture or $100 - \%S$

DF = Dilution Factor

9.3 The percent solids is calculated as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight

WW = Sample weight (g) before drying

9.4 Significant Figures:

All results for GRO range concentrations are reported to two (2) significant figures.

10. Quality Assurance/Quality Control

10.1 Method Detection Limits:

Method Detection Limits required per Maine GRO are 10ug/L for waters, and 2.5 mg/Kg for soils. There is no annual requirement for the study in the document. These limits have been achieved and are entered into LIMS. According to the NELAC standard, chapter 5; tests which are not reported below the concentration of the lowest level of the initial calibration, are not required to perform annual MDL studies. Mitkem does not report GRO results below our reporting limits.

DoD QSM projects using GRO by SW846 8015 methodology require annual MDL studies, and quarterly LOD verifications.

Annually, the laboratory performs the Maine method's Initial Demonstration of Capability to generate acceptable accuracy and precision for this method, by preparing and analyzing 7 LCS for both water and soil. The recoveries and RPD are calculated and stored in the QA Office.

10.2 Quality Control:

10.2.1 Initial Calibration acceptance criteria:

The initial calibration for GRO and BFB is acceptable if the percent RSD of the RF does not exceed 20 %.

ICV analysis following the ICAL must meet the +/- 20% recovery criteria.

Initial Calibration acceptance criteria must be met before any samples, Lab Control Samples, Duplicate Matrix Spikes or Blanks are to be analyzed.

10.2.2 Continuing Calibration acceptance criteria:

The Continuing Calibration is deemed acceptable if the RF does not exceed those of the Initial Calibration for more than 20% for both GRO and BFB.

10.2.3 Surrogate recoveries:

The recovery of the system monitoring compound BFB in all samples, blanks, matrix spikes and matrix spike duplicates will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

The percent recovery control limits for BFB are set annually using control charts of in-house data points. See Mitkem SOP No. 80.0010 for details. Recovery of BFB in Method Blanks must be within the control limits. The annual limits can be found in the test information form in LIMS.

10.2.4 Method Blank:

Method Blanks are analyzed to determine the level of contamination associated with the processing and analysis of samples. One Method Blank is analyzed per batch of samples. A Method Blank is analyzed at a frequency of one for up to 20 samples of a similar matrix. Method Blanks are prepared as in Section 8.5.3.1.

Method Blanks (or Instrument/Method Blank) are analyzed immediately after the ICAL/CCV and immediately before the Lab Control Sample.

10.2.4.1 Acceptance criteria for Method Blanks:

- Recovery of BFB in Method Blanks must be within the control limits.

- The instrument blank area may be subtracted from the Method Blank prior to evaluation.
- The concentration of target compounds found in the Method Blank must be less than the reporting limit of 25µg/L.
- Method Blanks are also deemed acceptable as long as the concentration of GRO is less than 1/10th the amount measured in any sample.

For DoD QSM and Maine GRO projects, the concentration cannot exceed one half the reporting limits.

10.2.5 Laboratory Control Sample (LCS):

Laboratory Control Samples are analyzed to determine the accuracy associated with the processing and analysis of samples. The LCS is prepared from a different source than that of the calibration standards and is a gasoline product.

10.2.5.1 Preparation of LCS:

An aqueous matrix LCS is prepared by spiking 100µL of a gasoline spiking solution into a 40mL VOA vial with analyte-free water and 1µL of the BFB spiking solution will be added to 5ml of the spiked solution when the Autosampler takes the aliquot to the sparger tube. Soil matrix LCS are prepared by spiking 100µL of a gasoline spiking solution and 700 µL of P&T methanol into a 40mL VOA vial with analyte-free water and 1µL of the BFB spiking solution will be added to 5ml of the spiked solution when the Autosampler takes the aliquot to the sparger tube. The LCS is then analyzed using the same procedure for sample analysis.

10.2.5.2 Frequency of LCS:

One LCS is analyzed per batch of samples per matrix. An LCS is generated at a frequency of once every 20 samples. If an MS/MSD is not analyzed, an LCSD must also be analyzed.

10.2.5.3 Acceptance criteria for Laboratory Control Samples:

- Recovery of BFB in the LCS must be within the control limits.
- The instrument blank area may be subtracted from the LCS prior to evaluation.

- The percent recovery of GRO for LCS is 80-120 for either matrix per Maine Method 4.2.17.
- When an LCSD is run, the %RPD should be $\leq 20\%$.

10.2.6 Matrix Spikes and Matrix Spike Duplicates:

Matrix spikes and matrix spike duplicates may be analyzed to assess sample matrix effect with the processing and analysis of samples.

10.2.6.1 Preparation of Matrix Spikes and Matrix Spike Duplicates:

Aqueous matrix spikes are prepared by spiking 100 μ L of a gasoline spiking solution into a 40mL VOA vial with sample and 1 μ L of the BFB spiking solution will be added to 5ml of the spiked sample when the Autosampler takes the aliquot to the sparger tube. Insure soil samples have a total methanol volume of 800 μ L of methanol in a 40ml of Vial with analyte-free water. The sample is then analyzed using the same procedure for sample analysis.

10.2.6.2 Frequency of Matrix Spikes and Matrix Spike Duplicates:

Duplicate matrix spikes are analyzed per batch of samples per matrix. Duplicate matrix spikes are generated at a frequency of at least once every 20 samples for samples of similar matrix.

10.2.6.3 Acceptance criteria for Duplicate Matrix Spikes:

Please note that the following criteria are advisory as accuracy and precision could be adversely affected by sample matrix. Recovery and precision outside of the limits of recovery should be documented in the project narrative to flag the possibility of sample matrix effects.

- The BFB recovery should fall within the in-house limits for the run to be considered valid, excluding matrix effect.
- The percent recovery limits of GRO for MS/MSD is 60-140% per Maine method 4.2.17.
- The instrument blank area may be subtracted from the MS/MSD prior to evaluation.
- % RPD for GRO recoveries should be $\leq 20\%$, where %RPD is determined as follows:

$$\%RPD = (R_{ms} - R_{msd}) * 200 / (R_{ms} + R_{msd})$$

10.2.7 Acceptance criteria for Samples:

Please note that the following criteria are advisory as accuracy and precision could be adversely affected by sample matrix. See corrective actions for samples in **section 12** for more information on sample matrix effect.

- The BFB recovery should fall within the in-house limits for the run to be considered valid, excluding matrix effect.
- The instrument blank area may be subtracted from the sample prior to GRO concentration quantitation.

10.3 All standards prepared from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

11.1 Reporting Limits:

The Reporting Limits (RL) for GRO is set to be equal to the lowest calibration standard that is still within the linear range of the calibration curve. The reporting limit for the entire range is based upon the summation of the reporting limits for each individual analyte in the component standard.

11.2 Data Review:

All data calculations must be reviewed 100% by the analyst or a peer. A final full technical review is done by the Lab Manager or another senior chemist, according to Mitkem's data review and validation procedures. GRO range integration is always checked for accuracy in all samples, Method Blanks, Laboratory Control Spikes, Matrix spikes, and Matrix spike duplicates. Since all positive results for GRO are manually integrated, the RIC showing the integration marks is an acceptable documentation of the "After" manual integration and no "Before" integration is required. Quantitation reports will mark the area with an "m" flag.

12. Corrective Action Procedures

12.1 Corrective Action for Continuing Calibration:

Repeat the Continuing Calibration if the 20% RF criteria is not met. Repeated failure to meet the criteria will necessitate performing a new Initial Calibration.

12.2 Corrective Action for Method Blanks:

Any Method Blank that fails to meet any of the criteria in **Section 10** must be reanalyzed. Any samples that are analyzed with the non-compliant Method Blank will be reanalyzed.

12.3 Corrective Action for LCS:

Any LCS that fails to meet any of the criteria in **section 10** must be reanalyzed.. Any samples analyzed with the non-compliant LCS will be reanalyzed.

12.4 Corrective Actions for Matrix Spikes and Matrix Spike Duplicates:

If the LCS is valid in the same batch, then no corrective action is required for Matrix spikes out of control limits, as long as there is reasonable consistency between the matrix spike and the matrix spike duplicate. In particular, if the sample contains significant GRO in comparison to the spike amount, calculation of percent recovery is typically difficult. If the sample contains no or low GRO in comparison to the spike amount, MS percent recovery should be within the QC limits.

12.5 Corrective Actions for Samples (also see section 10):

12.5.1 Hydrocarbon Concentration:

Corrective action involves diluting and re-analyzing samples if the concentration of hydrocarbons in the initial analysis exceeds the instrument calibration range. As GRO is a collection of many peaks, and the instrument is calibrated using selected individual peaks representing GRO, the height of the largest peak on the sample may not exceed the height of the largest component peak in the initial calibration.

12.5.2 Matrix Effect:

Samples will be re-analyzed for matrix effect if the percent recovery of BFB is outside the control limits listed above for samples. If both runs are out of control limits in a consistent fashion (both above or both below percent recovery, not one above and one below percent recovery), and if the percent recovery problem can not be determined by other factors (such as poor purging or degradation of the BFB surrogate), then both runs will be reported, and the matrix effect of the sample on the BFB recovery will be explained in the report narrative.

- 12.6 Any corrective action which must be performed for an Initial or Continuing Calibration, Method Blank, or Lab Control Sample as described in this document should be executed before other runs are performed, or the data for subsequent analyses are accepted.

Examples:

- If a continuing calibration does not pass acceptance criteria, then corrective action must be performed before any other samples are run.
- If an individual Initial Calibration level does not pass acceptance criteria, it may be run after other Initial Calibration levels, but it must be run before any other types of samples (Method Blanks, samples, etc.).

13. Health and Safety

All safety precautions as recommend by the instrument vendor should be followed. Analytical standards, samples and sample extracts may contain hazardous constituents and should be handled with care in a well-ventilated environment, and exposure to these compounds should be kept as low as reasonably possible.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Maine Health and Environmental Testing Laboratory. Modified GRO Method for Determining Gasoline Range Organics, Method 4.2.17, September 6th 1995.

U.S. Environmental Protection Agency. Nonhalogenated Organics Using Gas Chromatography/FID, Method 8015B, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, 1996.

U.S. Environmental Protection Agency. Nonhalogenated Organics Using Gas Chromatography/FID, Method 8015D, SW-846 Test Methods for Evaluating Solid Wastes on-line Edition, June 2003.

Quality Systems Manual for Environmental Laboratories Department of Defense, Final Version 4.1 April 2009

Attachments:

Attachment 1: A Component Standard chromatogram and integration report.

Attachment 2: A Gasoline Product Standard Chromatogram and integration report.

Attachment 3: Volatile GC/FID Instrument Run log.

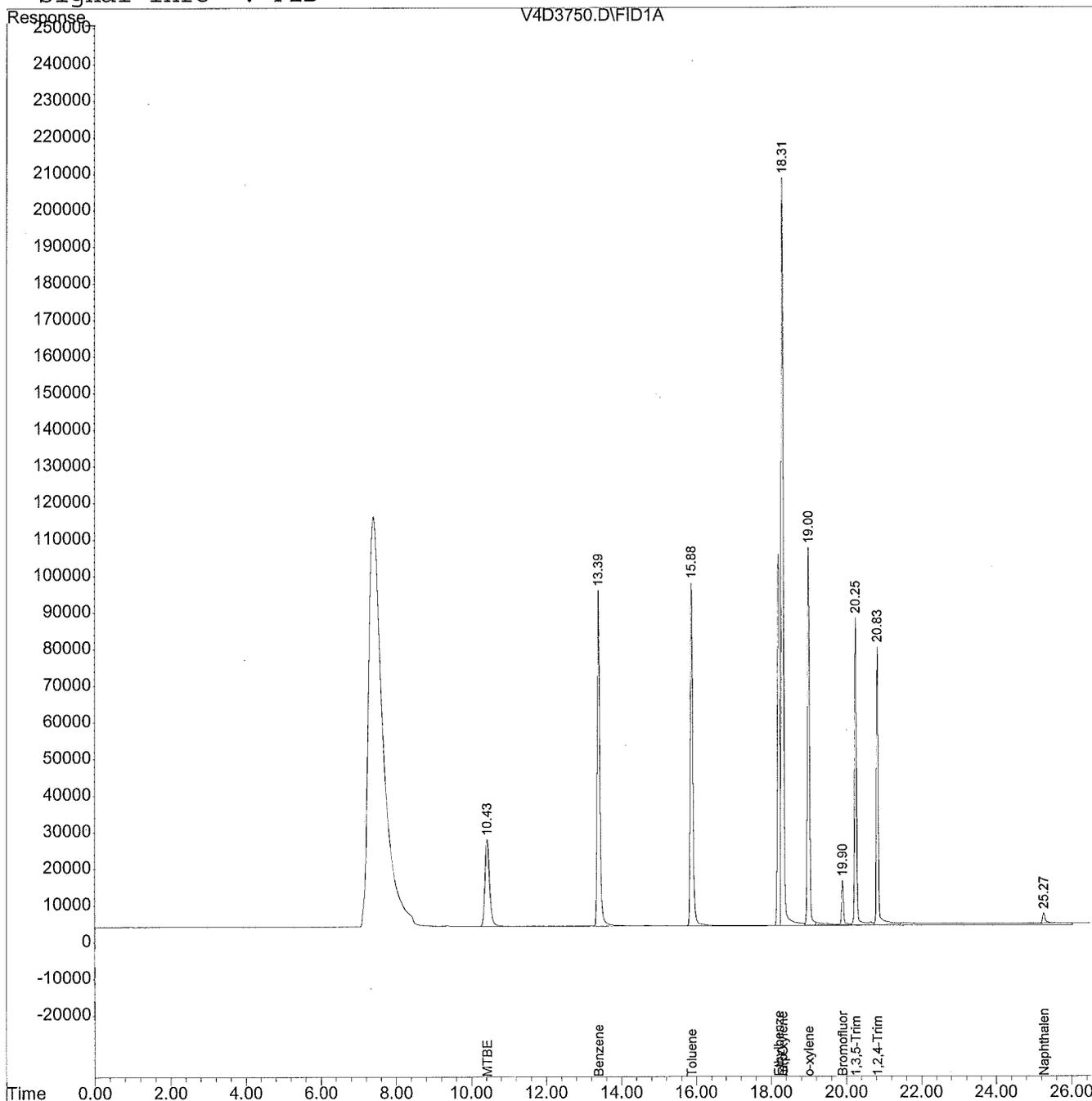
Attachment 1:

A Component Standard chromatogram and Integration report

Data File : O:\ORGANIC\VOA\V4.I\100607.B\V4D3750.D Vial: 4
 Acq On : 7 Jun 2010 15:27 Operator: sz
 Sample : 5ML,VSTD050V4,,, Inst : V4
 Misc : AQ Multiplr: 1.00
 IntFile : GRO.E
 Quant Time: Jun 8 14:13 2010 Quant Results File: GRO0418.RES

Quant Method : O:\ORGANIC\V...\GRO0418.M (Chemstation Integrator)
 Title : Method 8015 - Gasoline Range Organics
 Last Update : Wed Apr 28 15:19:46 2010
 Response via : Multiple Level Calibration
 DataAcq Meth : V4GRO.M

Volume Inj. :
 Signal Phase :
 Signal Info : FID



Data File : O:\ORGANIC\VOA\V4.I\100607.B\V4D3750.D Vial: 4
 Acq On : 7 Jun 2010 15:27 Operator: sz
 Sample : 5ML,VSTD050V4,,, Inst : V4
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 Last Update : Wed Apr 28 15:19:46 2010
 Response via : Initial Calibration
 DataAcq Meth : V4GRO.M

Volume Inj. :
 Signal Phase :
 Signal Info : FID

Compound	R.T.	Response	Conc Units
System Monitoring Compounds			
7) S Bromofluorobenzene	19.90	577786	17.965 ug/L m
Spiked Amount 20.000	Range 87 - 112	Recovery =	89.83%
Target Compounds			
1) T MTBE	10.43	2051246	49.416 ug/L
2) T Benzene	13.40	4484275	48.511 ug/L
3) T Toluene	15.88	4315442	52.237 ug/L
4) T Ethylbenzene	18.20	3927744	53.842 ug/L m
5) T m,p-xylene	18.31	7892486	97.595 ug/L m
6) T o-xylene	19.00	4117847	52.739 ug/L
8) T 1,3,5-Trimethylbenzene	20.25	3000081	44.098 ug/L
9) T 1,2,4-Trimethylbenzene	20.84	2650706	40.741 ug/L
10) T Naphthalene	25.27	225603	41.241 ug/L m
11) GRO	18.31	35254642	527.849 ug/L m

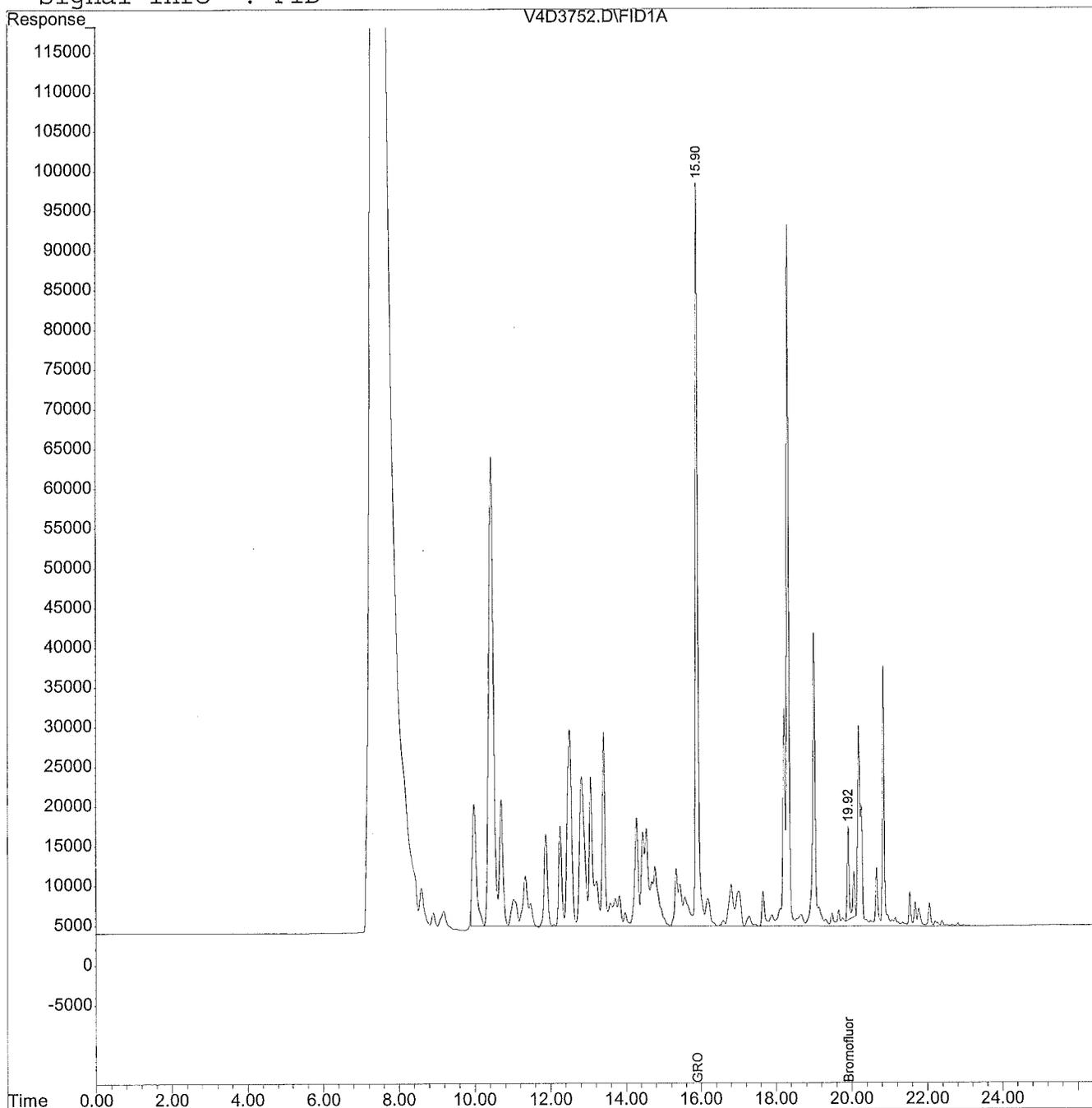
Attachment 2:

A Gasoline Product Standard Chromatogram and integration report

Data File : O:\ORGANIC\VOA\V4.I\100607.B\V4D3752.D Vial: 6
Acq On : 7 Jun 2010 16:40 Operator: sz
Sample : 5ML,LCS-52126,VLCSV4,52126, Inst : V4
Misc : AQ Multiplr: 1.00
IntFile : GRO.E
Quant Time: Jun 8 13:45 2010 Quant Results File: GRO0418.RES

Quant Method : O:\ORGANIC\V...\GRO0418.M (Chemstation Integrator)
Title : Method 8015 - Gasoline Range Organics
Last Update : Wed Apr 28 15:19:46 2010
Response via : Multiple Level Calibration
DataAcq Meth : V4GRO.M

Volume Inj. :
Signal Phase :
Signal Info : FID



Data File : O:\ORGANIC\VOA\V4.I\100607.B\V4D3752.D Vial: 6
 Acq On : 7 Jun 2010 16:40 Operator: sz
 Sample : 5ML,LCS-52126,VLCSV4,52126, Inst : V4
 Misc : AQ Multiplr: 1.00
 IntFile : GRO.E
 Quant Time: Jun 8 13:45 2010 Quant Results File: GRO0418.RES

Quant Method : O:\ORGANIC\V...\GRO0418.M (Chemstation Integrator)
 Title : Method 8015 - Gasoline Range Organics
 Last Update : Wed Apr 28 15:19:46 2010
 Response via : Initial Calibration
 DataAcq Meth : V4GRO.M

Volume Inj. :
 Signal Phase :
 Signal Info : FID

Compound	R.T.	Response	Conc	Units
System Monitoring Compounds				
7) S Bromofluorobenzene	19.92	586660	18.241	ug/L m
Spiked Amount 20.000	Range 87 - 112	Recovery	=	91.20%
Target Compounds				
1) T MTBE	0.00	0	N.D.	ug/L d
2) T Benzene	0.00	0	N.D.	ug/L d
3) T Toluene	0.00	0	N.D.	ug/L d
4) T Ethylbenzene	0.00	0	N.D.	ug/L d
5) T m,p-xylene	0.00	0	N.D.	ug/L d
6) T o-xylene	0.00	0	N.D.	ug/L d
8) T 1,3,5-Trimethylbenzene	0.00	0	N.D.	ug/L d
9) T 1,2,4-Trimethylbenzene	0.00	0	N.D.	ug/L d
10) T Naphthalene	0.00	0	N.D.	ug/L d
11) GRO	15.90	38920786	582.740	ug/L m

Attachment 3

Volatile GC/FID Instrument Run log

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

For

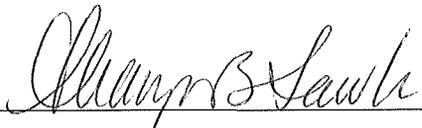
Total Petroleum Hydrocarbons by GC-FID
Using EPA SW-846 Method 8015/State Methods

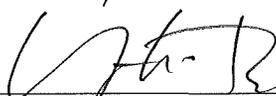
SOP No. 60.0050

Rev 11

Signature

Date

QA Director:  3/5/10

Lab Director:  3/8/10

Effective Date: 3/15/10

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.
STANDARD OPERATING PROCEDURE

for

**Extractable Total Petroleum Hydrocarbons by GC-FID
Using EPA SW-846 Methods 8015/ State Methods**

Rev 11

1. Scope and Application

- 1.1. This method describes the procedure for analysis of extractable total petroleum hydrocarbons (ETPH) in soil, sediment and aqueous samples (i.e. surface and ground water). The conditions are designed to measure the C₉ to C₃₆ range of hydrocarbons. This range represents the major components of a number of widely used petroleum products such as kerosene, jet and diesel fuels, No. 2 to No. 6 fuel oils and motor oil. This method is not used for quantitation of gasoline contamination because the major components of gasoline are not retained in the sample extraction and concentration procedure. This method is not intended for the analysis of potable water.
- 1.2. Some state or government agencies require various options included in this method. Where appropriate, these options are noted in separate boxes. This SOP describes Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM) protocols for analysis of Army, Navy and Air Force projects. Various state methods, such as Connecticut ETPH, New Jersey OQA-QAM-025, and Maine DRO are also included within the scope of this SOP.

2. Personnel Qualifications and Responsibilities

- 2.1. Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure. The Data Reviewer evaluates all laboratory reports for reasonableness of the results and signs the reports. The **QA Director** monitors

all aspects of the quality control program to insure the quality of the data generated passes an assessment of data accuracy and precision.

3. Summary of Procedure

- 3.1. A sample submitted for ETPH/DRO/TPH analysis is extracted with methylene chloride and dried over sodium sulfate. The extract is then concentrated in a Kuderna-Danish apparatus to a final volume of 1.0 ml.
- 3.2. The 1.0 ml extract is then analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram is collectively integrated within the C₉ through C₃₆ aliphatic hydrocarbon range. Average response factors determined using an aliphatic hydrocarbon standard mixture is used to calculate the collective concentrations of petroleum products eluting in the C₉ through C₃₆ aliphatic hydrocarbon range. Options for different hydrocarbon ranges are listed within this document.
- 3.3. This method is suitable for the analysis of waters, soils, wipes, and sediments. It can also be used as an identification procedure for oil and product samples when analyzed diluted in solvent.

4. Sample Preservation, Containers, Handling, and Storage

- 4.1. Aqueous samples are collected in 1-liter amber glass bottles with Teflon-lined screw caps.
- 4.2. Soil and sediment samples are collected in 4 oz. (120 ml) amber wide-mouth glass jars with Teflon-lined screw caps.
- 4.3. Aqueous samples may be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5 ml of 1:1 HCl to a liter sample. The use of alternative acids is permissible. Following collection and addition of acid, the sample must be cooled to 4°C.

Note: There are other methods for the analysis of petroleum hydrocarbons that allow for longer holding times if the sample is acid preserved. EPA Method 1664 (Oil & Grease and Total Petroleum Hydrocarbons) allows for a 28 day holding time if the sample is preserved to a pH <2. Massachusetts EPH method allows for a 14 day holding time if the sample is acid preserved. The 7-day holding time is a Maine DRO, Connecticut ETPH and generic SW-846 aqueous extractable holding time that applies to a wide range of methods, most of which are not amenable to acid preservation. Petroleum-specific methods allow for longer holding times as long as pH adjustment and refrigeration prevent the microbiological degradation of hydrocarbons. There is likely no loss of hydrocarbons in acid-preserved, refrigerated

samples between 7 and 28 days from collection. Aside from any technical or chemical justification, there may be important regulatory significance to the 7 day holding time.

- 4.4. Soil and sediment samples must be cooled to 4°C immediately after collection.
- 4.5. A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.
- 4.6. Aqueous samples must be extracted within 7 days of collection, except where noted **above**; and extracts must be analyzed within 40 days of extraction.
- 4.7. Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

NJ OQA-QAM-025 (section 9.2) requires a 14 day holding time VTSC (or 12 days VTSR) for both water and soils.

5. Interferences and Potential Problems

- 5.1. Washing all glassware with hot soapy water, then rinsing with warm tap water, acetone, and methylene chloride reduces method interferences.
- 5.2. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it will be followed by the analysis of a system solvent blank to check for cross-contamination.
- 5.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled. A silica gel SPE cleanup procedure may be used to overcome many of these interferences, but some samples may require additional cleanup approaches that are beyond the scope of this SOP.
- 5.4. Certain organic compounds not associated with the release of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantified as Total Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.
- 5.5. Fluoranthene, present in samples with significant PAH concentrations, can interfere with 1-Chlorooctadecane (COD), the Internal Standard.

5.6. This procedure will quantify those semivolatile portions of petroleum hydrocarbons amenable to solvent extraction/concentration/GC analysis. Portions of petroleum products beyond the semivolatile range (heavy tars) are not amenable to GC analysis, and will not be detected by this method. Volatile hydrocarbon products such as gasoline will have significant portions of the product lost during the extract concentration phase of the analysis, resulting in false-low results.

6. Equipment and Apparatus

6.1. Gas Chromatograph

- 6.1.1. Gas Chromatograph: Dual column HP6890 GC with programmable temperature. Chemstation and Enviroquant software are used to store and process data.
- 6.1.2. Capillary column: 30m x 0.32mm id x 0.25 μ m film thickness DB-5MS. Must allow for adequate resolution of alkanes C₉-C₃₆ in less than one hour. Must also be capable of resolving pristane from C17 and phytane from C18 in commercial diesel fuel per the State of Maine DRO method.
- 6.1.3. Flame Ionization Detector (FID).
- 6.1.4. Auto-sampler: HP 6890 Series Injector, 1ul injection volume.
- 6.1.5. Microsyringes: 10- μ l, 100- μ l, 250- μ l, 500- μ l, 1000- μ l.

7. Standards

7.1. Primary Standards

The standards used in this method are discussed below. *Please note that standards from other vendors may be used as long as the standards are of high purity (>96%) and traceable to reference materials.* This list includes several common hydrocarbon fuel standards. A subset of these fuels is analyzed with each initial calibration to aid in qualitative identification of petroleum products in samples. Other products may be analyzed from time-to-time depending on the needs of a particular project or client. The laboratory should have or have on order one complete set of unopened ampulated standards.

- 7.1.1. Accustandard Multi-State Hydrocarbon Window Defining Standard (Cat. # DRH-008S-R1) at 500 ug/mL is used as the primary calibration stock standard. This standard contains all the straight chain aliphatic hydrocarbons from C8 to C38, C40, Pristane and Phytane.

7.1.2. The following list of primary (ampulated) standards obtained from Restek are used for reference purposes when needed:

- MA EPH Aliphatics Standard at 1,000 ug/mL (Cat. # 31459).
- Connecticut Alkane Standard at 1,000 ug/mL (Cat. # 31614).
- #2 Diesel Fuel Composite Standard at 50,000 ug/mL (Cat. # 31258).
- #6 Fuel Oil Standard at 5,000 ug/mL (Cat. # 31218).
- JP-4 Military Jet Fuel at 5,000 ug/mL (Cat. # 31219).
- Kerosene Fuel Standard 5000 ug/mL (Cat. #31094).

7.1.3. The following list of primary (ampulated) standards obtained from Ultra Scientific are used for reference purposes when needed:

- EPH Aliphatic Hydrocarbon Standard at 1,000 ug/mL (Cat. # SMA-310).
- Unleaded Gasoline Standard at 5,000 ug/mL (Cat. # RGO-608).
- Fuel Oil #4 Solution at 5,000 ug/mL (Cat. # RGO-631).
- Fuel Oil #6 Solution at 50,000ug/mL (Cat. # RGO-652).
- SAE 10W30 Motor Oil Standard at 1,000 ug/mL (Cat #RGO-722).
- Jet Fuel A Standard at 5,000 ug/mL (Cat # RGO-671).
- Florida TRPH Standard at 500 ug/mL (Cat # SFL-601)

7.1.4. The 1-Chlorooctadecane (COD) Internal Standard is purchased as a neat concentration from Aldrich (Cat. #238368-25G). A 4000ug/mL solution is prepared.

7.1.5. 5- α Androstane Surrogate Standard material is purchased neat from Sigma (Cat. # A0887-1G). The O-Terphenyl (OTP) Surrogate Standard material is purchased neat from Aldrich (Cat. # T2800-25G), and the Chlorobenzene Standard is purchased neat from Ultra Scientific (used for NJ QAM projects only). These standards are used to prepare the extraction surrogates at 10,000ug/mL.

7.1.6. Other fuel identification standards (Diesel Fuel, Mineral Oil, Coal Tar, Lubricating Oil, Transmission Oil, etc) are purchased from commercial vendors such as fueling stations and automotive supply stores.

7.1.7. All of the standard information is recorded in the Primary Standard Logbook. All vials containing primary standards must be labeled according to the current version of Mitkem SOP 80.0001; Standard Preparation, Equivalency and Traceability.

7.1.8. The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. If no expiration date is given by the manufacturer, the unopened ampules are considered valid for two years.

Once opened, all primary standards have a 6 month expiration date. All expiration dates must be entered on the vial label. All primary standards are stored according to manufacturer's recommendation.

7.2. Working Standards

Using the primary standards listed above, the working standard volumes may be adjusted to make smaller or larger quantities of standards as long as the final working concentrations are the same. All standards made from a primary standard expire on or before the expiration date of the primary standard.

All of the following working standards are logged into the FID Working Standard Logbook, and labeled as FwymddX

where: FW = FID Working standard

yymdd = date the standard is prepared, in year/month/day format

X = the order the standard is prepared on that date, in alphabetical order

The standards are stored in amber containers under refrigeration at 4°C or below. All standards are stored in a separate location from the samples and/or extracts to minimize cross contamination.

The expiration dates for the working standards are six months from the date of preparation. All standard expiration dates are superseded by the primary standard's expiration date. All expiration dates must be entered on the vial label.

7.3. Stock Standards

- 7.3.1. The EPH aliphatic hydrocarbon standard consists of the 14 normal alkanes listed below in **Table 1**, and the surrogates. Prepare this stock using the Restek brand standards; the resultant concentration for each analyte (aliphatics and surrogate) will be 200 ug/mL each. This will be diluted to concentrations of 100, 50, 25 and 2.5 ug/mL to form the calibration curve.

Table 1: The n-alkane standard contains:

Carbon Number	Compound Name
9	n-Nonane
10	n-Decane
12	n-Dodecane
14	n-Tetradecane

16	n-Hexadecane
18	n-Octadecane
20	n-Eicosane
22	n-Docosane
24	n-Tetracosane
26	n-Hexacosane
28	n-Octacosane
32	<u>n- Dotriacontane</u>
36	n-Hexatriacontane
<u>Surrogates</u>	<u>5α-Androstane, OTP, chlorobenzene*</u>

7.3.2. The petroleum reference standard consists of a commercially available diesel fuel standard. Prepare this stock by dissolving 0.01g diesel fuel into methylene chloride in a 10ml volumetric flask.

7.4. Surrogate Standard

7.4.1. The surrogate standard is used to monitor the efficiency of sample extraction, chromatographic, and calibration systems.

7.4.2. The surrogate spiking solution is 5 α -Androstane, OTP and Chlorobenzene* at a concentration of 40 ug/mL each in methanol. Each sample, blank, and matrix spike is fortified with 1.0 ml of the surrogate spiking solution.

<u>*Chlorobenzene is a required surrogate for the NJ OQA-QAM-025 method.</u>

7.5. Internal Standard

7.5.1. Every 1.0 ml extract is spiked with 10 ul of the internal standard 1-Chlorooctadecane (COD) solution for a concentration of 40 ug/mL.

7.6. Matrix Spike Standard

7.6.1. The matrix spike solution is prepared from a different source as the calibration standards. The spiking solution consists of commercial diesel standard, and is prepared in acetone at a concentration of 5 mg/mL. The

samples selected as the matrix spike and the LCS are fortified with 1.0 ml of the matrix spiking solution.

8. Procedure

8.1. Sample Preparation: The following methods are routinely used to extract samples. The associated Mitkem SOPs are listed for reference.

- SW 3520 Continuous Liquid/Liquid Extraction, SOP No. 50.0050.
- SW 3510 Separatory Funnel Extraction, SOP No. 50.0051.
- SW 3550 Sonication Extraction, SOP No. 50.0052.
- SW 3540 Soxhlet Extraction, SOP No 50.0053.
- SW 3570 Microscale Solvent Extraction, SOP No. 50.0100.

8.2. Gas Chromatography

8.2.1. Gas Chromatographic Conditions (these may change slightly to achieve required QA/QC as the column degrades):

- Set oven temperature to 40°C for 0.5minutes, then ramp 14°C/min up to 305°C and hold for 8.07 minutes.
- Sample/auto-sampler injection is 1.0 µl.
- Carrier gas is hydrogen.
- Carrier Gas Flow: 2.8ml/min.
- Air: 400 ml /min.
- Hydrogen: 40 ml /min.
- Makeup gas flow: 40 ml /min.
- FID temperature: 320°C.
- Injection port temperature: 300°C.
- GC operated in splitless mode.
- Column splitless pressure: 20 psi for 0.50 min and a purge time of 0.55 min.
- Linear velocity: approximately 50 cm/sec.

8.2.2. GC Maintenance:

Maintenance is required if indicated by calibration verifications being outside of the QC limits. Maintenance is commonly indicated if the response of the C36 peak is significantly lower than the response of the C32 peak. As this analysis is able to extract high boiling point, non-volatile hydrocarbons and other compounds which are not amenable to chromatography; these compounds condense in the injection port and on the head of the GC column. The major effect of these compounds is to

reduce the response of the later-eluting hydrocarbons, as indicated by the C36 response.

- Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert.
- Break off the first few inches, up to one foot, of the injection port side of the column.
- Remove the column and solvent back flush according to the manufacturer's instructions.
- Bake out the column at 320°C. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.
- Record all routine maintenance in the associated instrument injection logbook. When more intensive maintenance is required it must be documented in the LIMS instrument maintenance log.

8.3. Retention Time Windows

- 8.3.1. Before establishing retention time windows, make sure the GC is within optimum operating conditions. Make three injections of the Aliphatic Hydrocarbon standard mixture throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.
- 8.3.2. Calculate the standard deviation of the three absolute retention times for each individual component in the Aliphatic Hydrocarbon Standard, the extraction surrogate, and the internal standard (where applicable).
- 8.3.3. Plus or minus three times the standard deviations of the absolute retention times for each standard should be used to define the retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.
- 8.3.4. In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.
- 8.3.5. The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. These data must be retained by the laboratory.

8.4. Calibration:

8.4.1. Calibrate the instrument using the internal standard procedure. The suggested internal standard for this method is 1-Chlorooctadecane (COD).

8.4.1.1. Using the stock standard from **section 7.3.1** (concentration 200 ug/mL per component), prepare 4 additional dilutions at 100, 50, 25 and 2.5 ug/mL. The lowest calibration standard must be at or below the Reporting Limit. The other concentrations must correspond to the expected range of concentrations found in real world samples or should define the working range of the detector.

For NJ OQA-QAM-025 projects, the working calibration standards are prepared using the SFL-601 primary standard, at concentrations of 1, 10, 20, 50 and 100 ug/mL. This calibration includes the 14 alkanes plus C₄₀, tetracontane.

8.4.1.2. Inject each calibration standard using the same technique that will be applied to the samples (e.g., 1 µl injection). Tabulate the peak height or peak area responses against the concentration of each individual compound and internal standard. Calculate response factors (RF) for each individual compound using *Equation 1* (**See Section 10**).

If the RF value over the working range is constant ($\leq 20\%$ RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations.

8.4.1.3. A collective response factor must also be established for the entire C₉ to C₃₆ hydrocarbon range. To calculate the collective RF, tabulate the summation of the peak areas of all components (all 14 normal alkanes) against the total mass injected using *Equation 2* (**See Section 10**). The collective RF is the average of the responses for the C₉ to C₃₆ individual compounds.

For NJ OQA-QAM-025 analysis, the collective RF is the average of the responses for the C₈ to C₄₀ individual compounds.

8.4.1.4. When a different hydrocarbon range is being quantified, the response factor will change to reflect that range.

For the Maine DRO analysis, the response factor includes only the C₁₀ to C₂₈ range hydrocarbons.

Note: The area of the extraction surrogates must **not be** included in the area of the Aliphatic Hydrocarbon range.

- 8.4.1.5. The initial calibration is followed by the same normal alkane standard at midlevel concentration from a second, independent source, to confirm that the calibration is valid (within $\pm 20\%$). Additionally the initial calibration should be followed by common fuel identification standards such as diesel, #4 fuel, #6 fuel and motor oil to ensure that the reference chromatograms for those standards reflect current operating conditions.

CT-ETPH requires that the individual RFs from the n-alkane standard also be evaluated against the average RF using the equation found in section 7.2.3 of the published CT method. This performance check is used to determine whether there is any significant sample introduction discrimination.

- 8.4.1.6. At a minimum, the RF must be verified on each working day, after every 20 samples per column, or after 12 hours, whichever is more frequent, by the injection of a mid-level calibration 50 ug/mL standard to verify instrument performance and linearity. If the relative percent difference (RPD) for TPH varies from the predicted response by more than $\pm 20\%$, as calculated using *Equation 3* (See **Section 10**), a new calibration curve must be prepared.

The **Maine DRO** method requires the GC to be able to resolve pristane from C₁₇ and phytane from C₁₈. The Maine DRO method requires that the resolution be 60% at the end of each sequence containing Maine samples. If this resolution cannot be demonstrated, the samples should be rerun. If this is not possible, the data must be qualified with the lack of resolution noted in the report narrative. Mitkem demonstrates this resolution in the closing CCV.

DoD QSM 4.1 requires calibration verification before sample analysis, after every 10 field samples and at the end of the sequence.

8.5. GC Analysis:

- 8.5.1. Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with blanks and QC samples. The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded.
- 8.5.2. Extracts are introduced into the gas chromatograph by direct injection.
- 8.5.3. Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window

for that day based on the first CCV. The daily retention time window equals the midpoint \pm three times the standard deviation determined in **Section 8.3**.

- 8.5.4. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation on a second GC column or by GC/MS may be necessary.
- 8.5.5. Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.
- 8.5.6. The ETPH range of interest is determined by the collective integration of all peaks eluting between specified range "marker" compounds.

For **CT-ETPH** and TPH analyses, the hydrocarbon range of interest is defined as all resolved and unresolved peaks eluting between C₉ and C₃₆ inclusive.

For **NJ OQA-QAM-025** analysis, the hydrocarbon range of interest is defined as all resolved and unresolved peaks eluting between C₈ and C₄₀ inclusive.

For the State of Maine and other Diesel Range Organic analyses, the hydrocarbon range of interest is defined as all resolved and unresolved peaks eluting between C₁₀ and C₂₈ inclusive.

- 8.5.7. When quantifying on a peak area basis by internal calibration, collective peak area integration must be from baseline (i.e., must include the unresolved complex mixture "hump" areas). For the integration of individual target analytes, surrogate compounds, and internal standards, a valley-to-valley approach should typically be used. Manual integrations are common for TPH/DRO analyses. Hardcopy of Before and After integrations are required for several programs including DoD QSM4.1.

8.6. Calculations

8.6.1. Internal Standard Calibration:

The concentration of each analyte and hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range

injected, from the peak response, based upon the analyte/internal-standard response ratio.

8.6.1.1. Aqueous samples:

The general equation to determine the concentration of a specific analyte or hydrocarbon range in aqueous samples is provided in *Equation 4 (See Section 10)*.

8.6.1.2. Non-aqueous samples:

The general equation to determine the concentration of a specific analyte or hydrocarbon range in soil or sediment samples is provided in *Equation 5 (See Section 10)*.

8.6.2. External Calibration Method:

In most circumstances where the internal standard cannot be resolved from the analytes of interest, samples are diluted and re-analyzed. This increases the relative proportion of internal standard to the unknown petroleum product so the internal standard peak can be more easily identified. There are circumstances where the external quantification approach may be used. This typically occurs when analyzing #6 Fuel. The Response Factor (*RF*) for external standard quantification is determined using *Equation 6 (See Section 10)*.

%RSD is calculated using *Equations 2 and 3 (See Section 10)*.

8.7. Data Upload to LIMS

Once the QC and samples have completed running they are quantified by uploading them to the LIMS System. On-column concentrations are obtained in the EnviroQuant software. These on-column concentrations are uploaded and adjusted by preparation, percent moisture and dilution factors within the LIMS to generate a final reportable result.

9. Quality Assurance/Quality Control

See **Attachments 2 and 3** for method specific QA requirements and general corrective action procedures.

9.1. The laboratory is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with

established performance criteria to determine if the results meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

- 9.2. A system solvent blank must be run after a sample suspected of being highly contaminated to determine if sample carryover has occurred.
- 9.3. At a minimum, for each analytical batch (up to 20 samples), a Method Blank (MB) and Laboratory Control Sample (LCS) must be run. A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) should be analyzed as well. If insufficient sample to perform MS/MSD analysis is provided, a duplicate LCS (LCSD) is analyzed. A Calibration Check Standard must be run after every 20 samples, or 12 hours (whichever is more frequent). The method blank and spiked samples must be carried through all stages of the sample preparation and measurement process.

9.3.1. A typical sequence would proceed as follows:

- (1) Continuing Calibration Verification (CCV)
- (2) Laboratory Method Blank
- (3) Laboratory Control Sample
- (4) Field Samples
- (5) Matrix Spike/Duplicate
- (6) Continuing Calibration Verification (CCV)* also used to demonstrate resolution for Maine DRO.

* NJ OQA-QAM-025 requires a calibration blank(CCB) be run after each CCV. DoD QSM requires CCV be run after a maximum of 10 field samples.

- 9.3.2. Control charts can be viewed in the LIMS which plot both LCS recoveries and surrogate standard recoveries as a function of time. At a minimum, when surrogate recovery from a clean soil sample, blank, or QC sample is out of control check the calculations to locate possible errors; check the fortifying solution for degradation and changes in instrument performance. If the cause cannot be determined, reanalyze the sample. Surrogate recoveries from clean water samples are often relatively low, with the method blank often having the lowest recovery for the entire batch. The extraction efficiency of the surrogate is lower in DI water than in samples containing minerals. Several methods offer the option of adding salt to improve extraction efficiency for aqueous samples.
- 9.3.3. In the case when the sample or MS/MSD contains petroleum product and surrogate recovery is outside the control limits, evaluate the data for validity. Resolved or unresolved hydrocarbons eluting in the same range

of the surrogate can interfere with the surrogate recovery. Re-extraction or reanalysis may not be required if calibration verifications indicate proper instrument performance, but it is important to contact the Supervisor or Operations Manager to confirm this.

9.4. Minimum Instrument QC

9.4.1. The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest. See **Section 8.4.** regarding Maine DRO resolution requirements.

9.4.1.1. The n-nonane (n-C₉) peak must be adequately resolved from the solvent front of the chromatographic run.

9.4.1.2. The surrogate and internal standards must be adequately resolved from any individual components in the Aliphatic Hydrocarbon standard.

9.4.1.3. All peaks of interest from the Aliphatic Hydrocarbon standard must be adequately resolved to baseline.

9.4.1.4. Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See **Section 8.3**)

9.4.1.5. Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the percent relative standard deviation (%RSD) over the working range of the curve is less than or equal to 20%.

9.4.1.6. The linear range of the aliphatic hydrocarbon standard should be established based upon peak height. The upper limit of the linear range is the peak height of the highest calibration standard. Samples containing peaks greater than the height of the highest calibration standard must be reanalyzed at dilution. Samples where the internal standard cannot be resolved must also be diluted, or quantified using the external standard procedure.

9.5. Initial and Periodic Method QC Demonstrations

The procedures specified in **Section 9.5.1 and 9.5.2** must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to significant

changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method or operational problems.

9.5.1. Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate acidified reagent water blanks and four clean sand blanks spiked with diesel fuel or the n-alkane mix at approximately 5 ppm. These are batched in LIMS as IPANDA1 through IPANDA4.

9.5.1.1. Analyze each replicate in accordance with the procedures described in **Section 8**.

9.5.1.2. Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte/product, and the precision (as %RSD) of the measurement for each analyte.

9.5.1.2.1. When using the n-alkane mix approach, the mean accuracy of each individual hydrocarbon, excluding C₃₆, expressed as a percentage of the true value, must be between 40% and 140 %. Poorer recoveries may be experienced for the C₃₆ standard. Additionally, C₉ may have recoveries down to 30% due to losses during sample concentration. For each analyte, the %RSD must be less than or equal to 25%.

9.5.1.2.2. When using the diesel fuel spike mix approach, the mean accuracy of the total diesel product, expressed as a percentage of the true value, must be between 60% and 140 %, and the %RSD must be less than or equal to 25%.

9.5.1.3. If desired, the Accuracy and Precision evaluation may be combined with the MDL evaluation specified in **section 9.5.2**.

9.5.2. Method Detection Limits for Diesel Range Organics

Analyze a minimum of seven replicate reagent water blanks and seven clean sand blanks which have been fortified with diesel fuel at 3 to 5 times the estimated or calculated Detection Limit, or at the reporting limit. Typical diesel fuel spiking concentrations are 0.1 mg/L for water, and 10 mg/kg for soil. Extract and analyze each replicate according to the procedures in **section 8**. Calculate the Method Detection Limit (MDL), using the procedure described in Mitkem SOP No. 80.0005 Determination of MDL. The MDL for TPH analysis does not have any particular significance except as a demonstration of capability. Sample results are never reported below the concentration of the lowest standard in the initial

calibration. After the MDL is determined, a verification check at 2-3times the MDL is run. The concentration of the verification check is referred to as an LOD.

DoD QSM4.1 requires establishing an MDL and LOD, and performing quarterly LOD verifications for method 8015 (DRO).

- 9.5.2.1. Water MDL are determined by extracting 7 to 10 replicates of 1-L acidified reagent water blanks spiked with surrogates and the petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of ~100 ug/mL. Spike a 1-L reagent water blank with 1.0 ml of this spiking solution.
- 9.5.2.2. Soil MDL are determined by extracting 7-10 replicates of 30-g sand blanks spiked with surrogates and petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of ~100 ug/mL. Spike 10g of clean sand blanks with 1.0 ml of this spiking solution.
- 9.5.2.3. The mean recovery of the summation of all hydrocarbon ranges (including target analytes), expressed as a percentage of the true value of the petroleum reference standard, should be between 40% and 140%.

9.6. On-going Method QC Demonstrations

- 9.6.1. Each sample, blank, and LCS must be spiked with the surrogate spiking solution. Surrogate recoveries must be within the range determined annually by the QC department (**Section 9.3.2**) or as set in the analytical method. Recoveries outside this range must be noted and discussed on the data report form. Recoveries outside of the acceptance range are common for samples containing interfering peaks, or for clean water samples with low mineral content that exhibit low extraction efficiency. This is especially true for 5 α -Androstane in water samples. Salting the water prior to spiking may help with the extraction efficiency. This surrogate should be evaluated using advisory limits due to its problematic characteristics.
- 9.6.2. At a minimum, with every batch of 20 samples or less the lab must analyze the following:
 - 9.6.2.1. **Continuing Calibration Verification Standard-** A midlevel calibration standard from the same stock solution used to develop the calibration curve must be analyzed prior to sample analysis to verify

the calibration state of the instrument. The CCV will be analyzed at a frequency of every 20 samples or every 12 hours, whichever is sooner. If the relative percent difference (RPD) for the collective TPH response factor or the surrogate varies from the predicted response by more than 20% new calibration curve must be prepared. For Maine DRO analysis, this standard must be evaluated at the end of each sequence to demonstrate acceptable resolution.

CT-ETPH: Requires a 30% D

ME DRO: Requires a 20%D

- 9.6.2.2. **Laboratory Method Blank-** A water or soil Laboratory Method Blank is prepared by fortifying a reagent water or clean sand blank with 1.0 ml of the surrogate spiking solution. TPH detected within the retention time window of any analyte or range of interest above the Reporting Limit must be noted on the data report form.
- 9.6.2.3. **Laboratory Control Sample-** A laboratory control sample is prepared by fortifying a reagent water or clean sand blank with 1.0ml of the surrogate spiking solution and 1.0 ml of the matrix spiking solution.

ME DRO LCS/LCSD recovery limits are set at 60-140, with a 20%RPD.

CT ETPH LCS/LCSD recovery limits are set at 60-140.

NJ OQA-QAM-025 LCS recovery limits are set at 70-120.

- 9.6.2.4. **Matrix Spike/Duplicate-** The water or soil MS/MSD is prepared by fortifying an actual water or soil sample with 1.0 ml of surrogate spiking solution and 1.0 ml of matrix spiking solution. The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations. The corrected concentrations of each analyte should be within the same recovery range as the LCS. The %RPD of the duplicates should be less than 50%.

CT ETPH and ME DRO MS/MSD limits are set at 50-150. NJ OQA-

QAM-025 MS/MSD recovery limits are initially set at 70-130.

- 9.6.3. If any of the performance standards specified in this section are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those

that are out of control must be rerun. If this is not possible, the issue must be discussed in the report narrative.

10. Data Reduction and Calculations

- *Equation 1: Individual Compound Response Factor:*

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \text{ or no IS } RF = \frac{(A_s)}{(C_s)}$$

where,

A_s = Response for the analyte to be measured.

C_{is} = Concentration of the internal standard, ng/ μ l.

A_{is} = Response for the internal standard.

C_s = Concentration of the analyte to be measured, ng/ μ l.

- *Equation 2: Range Response Factor:*

$$\text{Range } RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \text{ or no IS } \text{Range } RF = \frac{(A_s)}{(C_s)}$$

where,

A_s = Summation of peak areas of component standards.

C_{is} = Concentration of internal standard, ng/ μ l.

A_{is} = Response for internal standard.

C_s = Total mass concentration of injected standards, ng/ μ l.

- *Equation 3: Relative Percent Difference (RPD):*

$$RPD = \frac{R_2 - R_1}{(R_1 + R_2) / 2} \times 100\%$$

where,

R_1 = Calibration Factor from calibration curve.

R_2 = Calibration Factor from calibration check.

• Equation 4: Concentration in Aqueous Samples:

$$\text{Concentration } (\mu\text{g} / \text{L}) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(V_s)} \text{ or no IS}$$

$$\text{Concentration } (\mu\text{g} / \text{L}) = \frac{(A_x)(D)}{(RF)(V_s)}$$

where,

A_x = Response of the analyte, hydrocarbon range/TPH being measured, units may be in area counts or in peak height.

C_{is} = Amount of internal standard added to extract, ng.

D = Dilution factor, if a dilution was made on the sample prior to analysis. If no dilution was made, $D = 1$, dimensionless.

A_{is} = Response of the internal standard, units same as A_x .

RF = Response factor for analyte or hydrocarbon range/TPH, dimensionless.

V_s = Volume of aqueous sample extracted, ml.

• Equation 5: Concentration in Non-Aqueous Samples:

$$\text{Concentration } (\mu\text{g} / \text{Kg}) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(W_d)} \text{ or no IS}$$

$$\text{Concentration } (\mu\text{g} / \text{Kg}) = \frac{(A_x)(D)}{(RF)(W_d)}$$

where,

W_d = Dry weight of sample extracted, g. (See Equations 7 – 9).

A_x , C_{is} , D , A_{is} , and RF have the same definition as for aqueous samples.

• Equation 6: Response Factor (RF) for external standard quantification :

$$RF = \frac{A_x}{C_x}$$

where,

A_x = peak area of the Fuel Oil to be measured.

C_x = Concentration of the Fuel Oil to be measured.

• *Equation 7: % Moisture:*

$$\% \text{ Moisture} = \frac{\text{g sample} - \text{g dry sample}}{\text{g sample}} \times 100\%$$

• *Equation 8: % Dry Solids:*

$$\% \text{ Dry Solids} = 100 - \% \text{ Moisture}$$

• *Equation 9: Dry Weight:*

$$W_d \text{ (g)} = (\% \text{ Dry Solids}/100) (\text{g of extracted sample})$$

11. Data Validation and Reporting

11.1. Calibration

Using the internal calibration procedure (**Section 8.4.1**) calibrate the GC as follows:

- 11.1.1. Calculate an RF for the individual normal alkane compounds that comprise the aliphatic hydrocarbon standard.
- 11.1.2. Calculate an RF for each surrogate.
- 11.1.3. Calculate a collective RF for the total mass concentration of the C₉-C₃₆ Aliphatic Hydrocarbons (or appropriate range). Tabulate the summation of the peak areas of all component standards against the total mass injected. Do not include the surrogates.

11.2. Sample Analysis

- 11.2.1. Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for starting alkane and 0.1 minutes after the RT for the closing alkane :

11.2.1.1. The CT ETPH C₉ to C₃₆ range is calibrated using the sum of C₉, C₁₀, C₁₂, C₁₄, C₁₆, C₁₈, C₂₀, C₂₂, C₂₄, C₂₆, C₂₈, C₃₀, C₃₂ and C₃₆ (14 components). Since the C₃₆ peak often tails a bit, we will often go .15 to .2 min past the C₃₆ RT to include the entire C₃₆ area – this is done on a case by case basis. These analytes are noted on the calibration summary and the quant report with a peak type "T".

11.2.1.2. The DRO (Maine Method) C₁₀ to C₂₈ range is calibrated using the sum of C₁₀, C₁₂, C₁₄, C₁₆, C₁₈, C₂₀, C₂₂, C₂₄, C₂₆ and C₂₈ (10 components). These analytes are noted on the calibration summary and the quant report with a peak type "D".

11.2.1.3. The NJ OQA-QAM-025 TPH C₈ to C₄₀ range is calibrated using the sum of C₈, C₁₂, C₁₄, C₁₆, C₁₈, C₂₀, C₂₂, C₂₄, C₂₆, C₂₈, C₃₂, C₃₄, C₃₆ and C₄₀ (14 components). Since the C₃₆ peak often tails a bit, we will often go .15 to .2 min past the C₃₆ RT to include the entire C₃₆ area – this is done on a case by case basis. These analytes are noted on the calibration summary and the quant report with a peak type "N".

11.2.2. Determine the peak area count for the sample surrogate standards and internal standard. Subtract these values from the collective area count value within the appropriate hydrocarbon range.

11.2.3. Using the equations contained in **Section 10** or linear regression analysis, calculate the collective concentrations of all resolved and unresolved peaks within the range of interest, and individual concentrations of any sample surrogates and internal standard.

11.3. Reporting Limits:

The Reporting Limits (RL) for individual analytes are set to be equal to the lowest calibration standard that is still within the linear range of the calibration curve, assuming 100% extraction of the sample matrix. The reporting limit for the entire range is based upon the summation of the reporting limit for each individual analyte. Reporting Limits (or PQL) are listed in LIMS under the Test Information section of the individual testcode.

11.4. Data Review:

All data calculations must be reviewed 100% by the analyst or a peer. A final full technical review is done by the Lab Manager or another senior chemist, according to Mitkem's data review and validation procedures.

12. Corrective Action Procedures

All out-of-control situations must immediately be reported to the department supervisor or the QA Director. Out-of-control situations must be recorded in the LIMS Corrective Action Log and documented the situation returned to control as described in the SOP No. 80.0007 Corrective Action Procedures.

13. Health and Safety

The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. All Lab Analysts must refer to the Material Safety Data Sheets (MSDS) prior to handling any unfamiliar reagents.

14. Waste Management, Pollution Prevention, Definitions and Acronyms

Please see Section 20.0 of Mitkem's current Quality Assurance Plan.

15. References

SW-846 Method 8015B-Modified for Diesel Range Organics, Revision 3, December 1996.

SW-846 Method 8015D-Nonhalogenated Organics using GC/FID. Revision 4, June 2003.

Maine Health and Environmental Testing Laboratory. Modified GRO Method for Determining Diesel Range Organics, Method 4.2.17, September 6th 1995.

Connecticut Extractable Total Petroleum Hydrocarbons , Environmental Research Institute, University of Connecticut, March, 1999.

Department of Defense, Quality Systems Manual for Environmental Laboratories. Final Version 4.1, April 2009.

NJ OQA-QAM-025 -02/08 Quantitation of Semivolatile Petroleum Products in Water, Soil, Sediment and Sludge. Revision 7, 02/25/08.

Attachments:

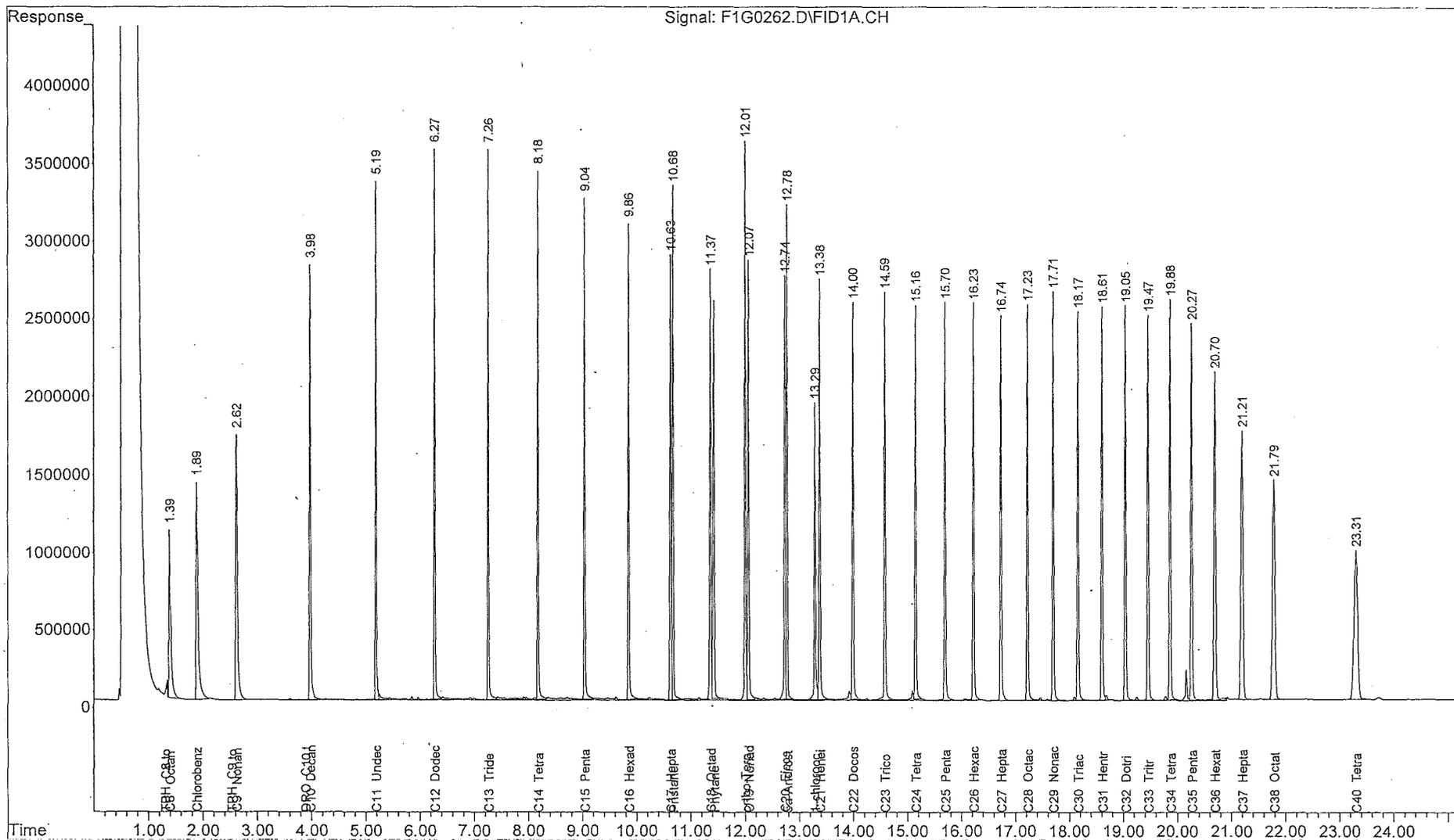
1. **Attachment 1:** TPH Component Standard Chromatogram.
2. **Attachment 2:** Method Specific QA Requirements.
3. **Attachment 3:** Corrective Action and Documentation for Method TPH (GC/FID).

Attachment 1

TPH Component Standard Chromatogram

Data File: O:\F1.I\090128.B\F1G0262.D
 Signal(s) : FID1A.CH
 Acq On : 28 Jan 2009 9:36 am
 Operator : TM
 Sample : ,FSTD0501S,FSTD0501S,,,
 Misc : | TPH CCAL L3 50 PPM
 ALS Vial : 1 Sample Multiplier: 1

Quant Time: Jan 29 12:29:26 2009
 Quant Method : O:\F1.I\QMETHODS\TPH1112F.M
 Quant Title : TPH, ETPH, DRO, Fuel ID, ORO
 Response via : Initial Calibration



Attachment 2

QA Criteria and Method Quality Objectives for Method SW8015 for DoD

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 15\%$ for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq 15\%$ for both DDT and Endrin.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$; Option 2: linear least squares regression: $r \geq 0.995$; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL; <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within $\pm 20\%$ of expected value from the ICAL; HPLC methods: All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-2. Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (Methods 8011, 8015, 8021, 8070, 8081, 8082, 8121, 8141, 8151, 8310, 8330, and 8330A) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD \leq 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 3

Corrective Action and Documentation for Method TPH (GC-FID)

ITEM #	OCCURRENCE	ACTION	DOCUMENTATION
1	RSD% of target compounds such as 5- α androstane, OTP and diesel and other petroleum products for initial calibrations exceeds 20%.	<ul style="list-style-type: none"> • Full maintenance will be done and a new curve will be run. • If RSD% for this new curve is still not ok, five levels fresh standards will be prepared and run. • If still not good, check chromatograph if column bleeding occurs. If column bleeding does happen, the old column will be replaced with a new one. • If still not good, make a service call. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned. • Any maintenance will go to LIMS maintenance logbook.
2	D% for initial calibration verification (ICV) exceeds 20%.	<ul style="list-style-type: none"> • Check if ICV standard is right one. • Check the preparation of ICV standard. • Check preparations for five level initial calibration standards if the ICV standard is ok. • Re-analyze if necessary. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
3	D% for continuing calibration verification (CCV) exceeds 20%.	<ul style="list-style-type: none"> • If D% is out but positive, and target compounds are lower than the reporting limits, no further action is required. • If D% is out and also negative, following further actions are needed: • Maintenance will be done including replacing septum, liner, gold seal, and trimming the column (full maintenance). • Check if CCV standard is evaporated, and if it does, make a fresh CCV standard. • If still not good, a new curve will be analyzed. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned. • Any maintenance will go to

			maintenance logbook.
4	Method blank contains petroleum products that are higher than reporting limits.	<ul style="list-style-type: none"> • This method blank will be re-analyzed to check if contamination is carried over. • Check if the blank and the other sample are switched. • After the blank contamination is confirmed, the samples that are related to this blank will be re-extracted and re-analyzed. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
5	Diesel or other spiked petroleum products in LCS are out of control limits.	<ul style="list-style-type: none"> • Check if it is double spiked. If it is, no further action is needed. • No further action is needed if LCS recovery is positive and the samples that are related to this LCS are lower than the reporting limits. • LCS will be re-analyzed, and if the result is still not ok, re-extraction and re-analysis for the samples that are related to this LCS will be done. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
6	Surrogate recovery in method blank or LCS is out of control limits.	<ul style="list-style-type: none"> • No further action is needed if surrogate recovery is higher than the upper limit and there are no target hydrocarbons detected in the related samples. • The method blank or LCS will be re-analyzed if surrogate recovery is lower than the lower value of control limits. If the results of re-analysis are still the same, the samples that are related to this blank or LCS will be re-extracted and re-analyzed. • The method blank or LCS will be re-analyzed if surrogate recovery is higher than the upper value of the control limits and the samples that are related to this blank or LCS are detected for target hydrocarbon products. If the result of re-analysis is the same, the batch will be re-extracted and reanalyzed. 	<ul style="list-style-type: none"> • This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.

7	Surrogate recoveries in samples are out of control limits.	<ul style="list-style-type: none"> No further action is needed if surrogate recovery is higher than the upper limit and there are no target hydrocarbons detected in the samples. The extracts will be re-analyzed if surrogates are out of limits and target petroleum products are detected in these samples. If the re-analysis results are the same, the affected samples will be re-extracted and re-analyzed. 	<ul style="list-style-type: none"> This will be noted in the instrument run logbook. The information will be noted on the package checklist for documentation in the project narrative. This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
8	Matrix spike (MS) recovery is out of QC range.	<ul style="list-style-type: none"> If duplicate spike (MSD) shows the same effect, it is generally matrix effect, and no further action is needed except documentation. However, frequent failures will require investigation to check if concentration of spiking standard is correct. 	<ul style="list-style-type: none"> This will be noted in the instrument run logbook. The information will be noted on the package checklist for documentation in the project narrative. This will be recorded in the LIMS Corrective Action Logbook and a CAR# assigned.
9	Relative Percent Difference (RPD%) for Matrix spike (MS) and Matrix Spike Duplicate (MSD) recoveries is out of QC range.	<ul style="list-style-type: none"> This limit is advisory. However, failures will require investigation such as checking if concentration of spiking standard is correct and spiking volume is consistent. Documentation is required. 	<ul style="list-style-type: none"> This will be noted in the instrument run logbook. The information will be noted on the package checklist for documentation in the project narrative. This will be recorded in the LIMS Corrective Action Logbook and a CAR# assigned.

**MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.**

STANDARD OPERATING PROCEDURE

for

**Determination of Metals in Water and Soil
by Inductively Coupled Argon Plasma Atomic Emission Spectrometry
using Method SW846 6010C**

Rev. 12

Signature

Date

QA Director:

Sharon B. Lamb

2/3/09

Lab Director:

Kim Chui

2/3/09

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

**Determination of Metals in Water and Soil
by Inductively Coupled Argon Plasma Atomic Emission Spectrometry
using Method SW846 6010C
Rev. 12**

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the elements listed in **Attachment 1**. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. **Section 8.1** provides the Method references for sample digestion procedures. See **LIMS Test Information/ Test/Limits** for analytes and their associated MDL/PQLs.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

- 3.1 Prior to analysis, samples must be digested using appropriate sample preparation methods.
- 3.2 The method measures element specific emitted light by optical spectrometry. The samples are nebulized and the resulting aerosol is transported to the plasma torch. The metals pass through the hot zone of the plasma, where they take up energy. Subsequently the metals pass through the cold zone (relatively) of the plasma where they give up the excess energy at element specific wavelength. The spectra are dispersed by a grating spectrometer, and the intensity of the emitted light is measured by a solid state photomultiplier. Background correction is required for trace element determination. 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 interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

4. Sample Preservation, Containers, Handling, and Storage

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For metals analysis by Method 6010C, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.
- 4.2 Soil samples are stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ until analyzed.
- 4.3 Sample holding time for metals analysis by method 6010C is 180 days from the date of sample collection for both water and soil.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICAP-AES.

- 5.1 Spectral interferences - Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena. The first of these can be compensated for by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. The second effect may require selection of an alternative wavelength. In addition one could select an alternate wavelength where interference is minimal or absent. The 4300DV and the 3100XL used at Mitkem have many spectral lines from which to choose. The third effect can usually be compensated by a background correction adjacent to the analyte line.
- 5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.
- 5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of

operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

- 5.4 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, or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.
- 5.5 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. If interference caused by a particular sample matrix is known, in many cases it can be circumvented. However, when the nature of the sample is unknown, following tests can be used to ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.
- 5.5.1 Dilution Test -If the analyte concentration is sufficiently high (minimally a factor of 10x above the method detection limit (MDL)), an analysis of a 1:5 dilution should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect could be suspected.
- 5.5.2 Post Digestion Spike Addition - If the matrix spike (pre-digestion) recovery falls outside of the control limits (75% - 125%), matrix interference is suggested. In this case an analyte spike is added to a portion of a prepared sample or its dilution at a level just below the mid-point of the calibration curve. Post digestion spikes should be recovered to within 80% - 120% of the known value. If not, a matrix effect should be suspected.
- 5.5.3 Comparison with alternative method analysis-when investigating a sample matrix, comparison tests may be performed with other analytical techniques, such as atomic absorption spectrometry, or ICP-mass spectrometry. This should only be done after consultation with the client.
- 5.5.4 Internal Standard Addition technique can be used (Refer to Section 4.4.2 of Method 6010C.)

6. Equipment and Apparatus

- 6.1 Inductively coupled argon plasma emission spectrophotometer (ICAP).

The ICAPs used at Mitkem are a Perkin-Elmer Model 4300DV and a 3100XL. The 4300DV is outfitted with an AS-93plus, 157-position autosampler and a high precision, three channel, peristaltic pump. The 3100XL is outfitted with an AS-91, 160-position

autosampler and a high precision, three channel peristaltic pump. Both ICPs have axial viewing capability, as compared to the more traditional radial viewing ICAP. Axial viewing provides greater sensitivity for all elements analyzed. The solid state detector is capable of analyzing at approximately 6000 wavelengths.

The built-in radio-frequency generator is FCC compliant.

The systems are computer controlled through a 32-Bit, Microsoft Windows NT operating system. This system allows for great flexibility in controlling the instrument.

The required high purity argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.1.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. Instrument detection limits, linear dynamic ranges, and interference effects are established for each analyte line used. All measurements must be in the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

6.1.2 Daily emissions of the highest standard for arsenic, copper, lead and selenium are recorded as a means to ensure the plasma is stable before analysis and also to chart potential problems especially with the power tube, sample introduction system, or RF generator. The normal range of the four analytes above are (the unit is intensity generated by instruments):

Optima 2 (3100 XL)	Optima 3 (4300 DV)
As 2,000 - 3,000	As 2,000 - 2,500
Cu 600,000 - 900,000	Cu 400,000 - 600,000
Pb 12,000 - 20,000	Pb 4,000 - 8,000
Se 2,000 - 4,000	Se 1,800 - 2,400

6.1.3 For analysis of normal environmental samples use the following standard operating conditions:

Wattage: 1450 – 1500
Argon flow rate (L/min): 15
Nebulizer flow rate (L/min): 0.55
Sample flow rate (mL/min): 2
Rinse time (sec): 60
Read delay (sec): 60

6.1.4 For regular day-to-day ICAP measurements, the plasma need not be optimized prior to analysis. The plasma needs to be optimized each time a major change occurs in instrument configuration, such as replacement of the torch and/or maintenance, unless response is still within normal ranges. Responses and alignment are routinely checked after torch cleaning or replacement with the analysis of a 1 mg/L Mn solution for axial view calibration and 10 mg/L Mn solution for radial view calibration. "Optimization" is a function of a series of procedures; refer to the Mitkem SOP 100.0006 for additional information on how to perform general maintenance and troubleshooting procedures.

6.1.5 To optimize the plasma, follow the instructions in the Optima Software Guide.

6.2 ICAP Instrument Maintenance:

6.2.1 Preventative Maintenance:

- Peristaltic pump tubing will be replaced every 16 hours of instrument time or sooner if memory effects are manifested.
- The plasma torch is cleaned using concentrated HNO₃ when torch and sample injector buildup is noted.
- The spray chamber and nebulizer are cleaned approximately every month as needed. Replacement is done when needed.
- Air filters are cleaned once every two weeks or as needed upon visual inspection.
- The RF coil, window and cone are cleaned every 2-3 weeks.
- The instrument undergoes extensive maintenance by a manufacturer's service engineer as needed.

6.2.2 Troubleshooting:

6.2.2.1 Sudden drop in CCV concentration occurs in a run:

- Check to see that sample introduction system (tubing) has not become disconnected.
- Check to see if a clog has occurred in the sample introduction system.
- Check CCV Standard to ensure it is not empty.
- Check room temperature to see if a fluctuation has occurred.

- Recalibrate and rerun if necessary.

6.2.2.2 CCVs drift up and down:

- Remake the CCV standard.
- Check room temperature to see if a fluctuation has occurred.
- If problem persists, call Perkin-Elmer. The problem is usually indicative of a power tube failure.

6.2.2.3 Plasma goes out during analysis:

- Sample is not reaching spray chamber (tubing came apart or there is a clog)
- High levels of salt in the sample caused plasma temperature changes.
- The sample injector is coated with high salt material and needs to be changed.
- The power tube is going (after all others fail repeatedly) and Perkin-Elmer must be called for service.

6.3 Glassware:

6.3.1 Class A volumetric flasks: 100 mL and 50 mL.

6.3.2 Class A volumetric pipettes:

6.3.3 10-100 μ L adjustable Eppendorf.

6.3.4 100-1000 μ L adjustable Eppendorf.

6.3.5 5mL adjustable Fisher Scientific.

6.3.6 100 μ L and 1mL fixed Wheaton.

6.3.7 10 mL Wheaton ICP tubes.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, Accustandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. *Please note that standards and reagents from other vendors could be*

used as long as the standards are of high purity (>96%0 and traceable to reference materials. All standards are labeled as instructed in Mitkem SOP # 80.0001 Standard Preparation, Equivalency and Traceability.

- 7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.
- 7.2 Nitric acid (conc.), HNO₃, Trace Grade, Fisher Scientific.
- 7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.
- 7.4 Mixed calibration standard solutions. Mixed calibration standards, obtained from High Purity Standards, come in three stock solutions. Calibration standards and QC solutions are made up on a daily basis.
 - 7.4.1 CLP CAL 1 consists of two solutions:
 - CLP CAL 1B contains silver only.
 - CLP CAL 1A contains all other elements other than antimony, arsenic, cadmium, lead, thallium, and selenium.
 - 7.4.2 CLP CAL 2 contains antimony only.
 - 7.4.3 CLP CAL 3 contains arsenic, cadmium, lead, thallium, and selenium.
- 7.5 High standard (**ICP Standard 1**):
 - Pipet 1mL HNO₃ (conc) and 1.5mL HCl (conc) into a 100mL volumetric flask.
 - Pipet 1mL each of CLP CAL 1A & 1B stock solution and 0.1mL each of intermediate CLP CAL 2 and 3 into the flask.
 - Pipet 0.1mL of Titanium single element standard into the flask.
 - Bring to volume with DI water.
- 7.6 Second (middle) standard (**ICP Standard 2**):
 - Pipet 5mL of a 1% HNO₃ and 1.5% HCl acid mixture into a 10 mL ICP tube.
 - Add 5mL of the ICP Standard 1.
- 7.7 Third (low) standard (**ICP standard 3**):

- Pipet 10mL of 1% HNO₃ and 1.5% HCl solution into a 10 mL ICP tube.
- Withdraw 100µL.
- Add 100µL of ICP Standard 1 and mix well.

See **LIMS Test Information/ Test/SPEC** for concentrations of the Initial Calibration Standards.

7.8 **Second source** ICV/CCV standards (CCV100XCONC and Antimony (Sb) Stock solutions) are obtained from Accustandard.

- Pipet 1mL of HNO₃ (conc.) and 1.5mL HCl (conc.) into a 100mL volumetric flask.
- Pipet 1.5mL (for ICV)of CCV100XCONC stock solution into the flask.
- Pipet 0.15mL of Antimony (Sb) Stock solution into the flask.
- Bring to volume with DI water.
- Transfer the solution to a 50mL plastic ICP tube.
- This standard is prepared as needed (in amounts consistent with the ratios above) usually every 1 – 2 days.

See **LIMS Test Information/ Test/SPEC** for analytes, concentrations and recovery limits for the ICV/CCV.

7.9 QC Standards ICSA and ICSB solutions are obtained from High Purity Standards.

ICSAB solution: In addition to the normal element composition of the ICSA solution, sodium, potassium, lead and selenium are added to the ICSAB solution. These stock solutions are also obtained from High Purity Standards.

- 7.9.1 Pipet 1mL of HNO₃ (conc.) and 1.5mL HCl into a 100mL volumetric flask.
- 7.9.2 Pipet 1mL of ICSB stock solution (ANALCS-R) into the flask.
- 7.9.3 Pipet 10mL of ICSA (CLP-INF-1) stock solution into the flask.
- 7.9.4 Pipet 0.5mL of the sodium/potassium stock solution (INFCS-5) into the flask.

7.9.5 Pipet 1mL of 45mg/L Pb intermediate standard solution into the flask.

7.9.5.1 The intermediate is prepared by adding 4.5mL of Pb at 1000mg/L (#100028-1) up to 100mL with 1%HNO₃ and 1.5% HCL acid solution in a volumetric flask.

7.9.6 Pipet 1mL of 45mg/L Se intermediate solution into the flask.

7.9.6.1 The Selenium intermediate standard is prepared by adding 4.5mL of Se at 1000mg/L (#031) up to 100mL with 1% HNO₃ and 1.5% HCL acid solution in a volumetric flask

7.9.7 Bring to volume with DI water.

7.9.8 Transfer the solution to a 10 mL ICP tube.

See **LIMS Test Information/ Test/SPEC** for concentrations of the ICSA and ICSAB.

7.10 Laboratory control samples (LCS) and Matrix spikes: The LCS standards for soils and waters are obtained from High Purity Standards.

- LCS/Spike standard 1 is prepared by adding 455uL of CLP-CV-1 to the digestion tube.
- LCS/Spike standard 2 and 3 are prepared by adding 45.5uL each of CLP-CV-2 and CLP-CV-3 to the digestion tube.

See **LIMS Test Information/ Test/SPEC** for final concentrations of the LCS/MS.

7.11 Linear Dynamic Range

The stock linear dynamic range (LDR) standards are purchased from Ultra Scientific, Inorganic Ventures and High Purity. The LDR is determined as the highest concentration of standard in which the determined value is within 10% of the true value.

- *LDR Standard I* is prepared by addition of 1 mL of Pb at 1000 mg/L, volumized to 10 ml with 1% HNO₃ and 1.5% HCl. Final concentration 100mg/L.
- *LDR Standard II* is prepared by addition of 5 mL of mix QCS-19 and 0.5mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 50 mg/L.

- *LDR Standard III* is prepared by addition of 2.5 mL of mix QCS-19 and 0.25 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 25 mg/L.
- *LDR Standard IV* is prepared by addition of 1.0 mL of mix QCS-19 and 0.1 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 10mg/L.
- *LDR Standard V* is prepared by addition of 0.5 mL of mix QCS-19 and 0.05ml of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 5mg/L.
- *LDR Standard VI* is prepared by addition of 0.5 mL of Al and 0.5ml of Mg at 10,000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 500mg/L.
- *LDR Standard VII* is prepared by addition of 3.0 mL of Fe at 1000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 300mg/L.
- *LDR Standard VIII* is prepared by addition of 0.5 mL of Ca at 10,000 mg/L, volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 500mg/L.
- *LDR Standard IX* is prepared by addition of 0.5 mL of mix INFCS-5 (High Purity) and volumized to 10 mL with 1% HNO₃ and 1.5% HCl. Final concentration 250mg/L.

8. Procedure

8.1 The methods in SW-846 for sample digestion or preparation are as follows:

- 8.1.1 Method 3005 , SOP No.100.0003, prepares ground water and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with hydrochloric and nitric acids prior to metal determination.
- 8.1.2 Method 3010 , SOP No.100.0003, prepares aqueous samples, mobility-procedure extracts and waste samples that contain suspended solids for total metal determination by ICP. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.
- 8.1.3 Method 3050 , SOP No.100.0104, prepares solid waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrochloric acid.

- 8.2 The wavelengths and background correction locations in the reference method can be substituted if they can provide the needed sensitivity and are corrected for spectral interferences. The analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

For each analyte there are a number of possible wavelengths at which analyses could be made. The wavelengths used were selected based on consultations with the ICP specialists at Perkin-Elmer and our own experience.

- 8.3 Background correction factors are obtained by aspirating a concentrated solution, usually about 200-500ppm, of the interfering element, and measuring the resulting concentrations at the wavelength used. The background correction factors for that interfering element are obtained by dividing the measured concentrations by the actual concentration. For example: by aspirating a 500ppm solution of aluminum, one measures a cadmium concentration of 0.010ppm. The aluminum correction factor for cadmium is $(0.010)/500 = 0.000020$. This means that if the aluminum concentration in a sample is, say, 120ppm, one must subtract $120 * 0.000020 = 0.0024\text{ppm}$ from the measured cadmium concentration in that sample.
- 8.4 Interelement Correction (IEC) factors are established quarterly (or biannually at a minimum), for the major salts: aluminum, calcium, iron, magnesium, and also for chromium, copper, manganese, nickel, thallium, titanium and vanadium. Correction factors should not exceed 20% difference from the previous IEC values.
- 8.5 Linear Dynamic Range is performed quarterly (or biannually at a minimum). Following instrument calibration, solutions with varying concentrations (**section 7.5.13**) of each analyte (LDR standards) are analyzed. The highest concentration, within $\pm 10\%$ of the true value, establishes the linear range.
- 8.6 Allow the plasma to become thermally stable before beginning the analyses. This usually requires at least 30 minutes. If the plasma is (for whatever reason) extinguished and needs to be re-lit, subsequent re-stabilization of the plasma takes only 15 minutes provided the plasma is re-lit immediately after it is extinguished.
- 8.7 The nebulizer flow rate used is constant for all aqueous sample extracts and needs not be reset prior to analysis.
- 8.8 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits (IDL). (See **Section 10** for IDL calculation)
- 8.9 The calibration curve consists of a blank (S0) and at least 3 calibration standards. The low concentration calibration standard concentration is less than or equal to the method reporting limit (MRL). Sample concentrations less than the lowest standard can only be reported after method modifications have taken place, or with estimated results flagging.

The highest concentration must be within the instrument's linear dynamic range. The standards are run in a sequence from high concentration to low concentration.

- 8.10 The minimum correlation coefficient for the calibration curve is 0.998. If a correlation coefficient less than 0.998 is obtained, the calibration must be repeated.
- 8.11 A mid-range ICV (Initial Calibration Verification) Standard from an independent source than the calibration standards) precedes the analysis of the samples. Recovery limits for the ICV are $\pm 10\%$ of the true value. If the ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. An ICV can be reanalyzed only once prior to corrective action.
- 8.11.1 A low level ICV (not required to be second source) is run to verify the lower level of calibration . Recovery limits for the low level ICV are + 30% of the true value. The low level ICV is the same concentration as the lowest standard in the initial calibration (S3), which is at the reporting limit. If the low level ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. A low level ICV can be reanalyzed only once prior to corrective action.
- 8.12 A CCV (Continuing Calibration Verification Standard) is analyzed after at least every tenth sample and at the end of the sample run. Recovery limits for the CCV are $\pm 10\%$ and in the case of failure for a particular element, all samples following the last acceptable CCV must be reanalyzed for that element. A CCV can be reanalyzed only once prior to corrective action.
- 8.13 The ICB is analyzed after the ICV and is of the same source as the calibration blank. The CCB (Continuing Calibration Blank) is analyzed after each CCV. Concentrations of any analytes detected must not exceed the MRL or corrective action, such as a single reanalysis and evaluation, must be taken. If the ICB/CCB still fails, reanalysis of all samples since the last valid ICB/CCB for that element is required.

DoD QSM: The acceptable absolute value of the ICB and CCB must be $< 2x$ MDL.

- 8.14 The ICS (ICSA and ICSAB) standards must be run at the beginning, every 8 hours and at the end of each analytical run.
- 8.14.1 The ICSA solution, containing the Al, Ca and Mg at 500mg/L, and Fe at 200mg/L, must be run at all wavelengths used for each analyte reported. The analytical results for those target analytes with MRLs $\leq 10\mu\text{g/L}$ shall fall within $\pm 2x$ MRL of the analyte's true value (the true value shall be zero unless otherwise stated). If the results for these analytes fall outside the $\pm 2x$ MRL window, check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. For analytes with a MRL $> 10 \mu\text{g/L}$, the ICSA results shall fall with \pm one MRL of the analytes true value (0).

DoD QSM: the absolute value of all non-spiked analytes < 2x MDL

- 8.14.2 The ICSAB contains Al, Ca, Mg and Fe at the same concentrations as the ICSA as well as all other analytes of interest. Recovery limits for the ICSAB must be within $\pm 20\%$ of the true value. If the ICSAB recoveries do not meet acceptance criteria, corrective action must be taken. Check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. If the ICSAB at the end of an analytical sequence fails for a particular element, all samples must be reanalyzed for that element.
- 8.15 After completion of the initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine. A 60 second rinse (rinse solution: 1% HNO₃ and 1.5% HCl) is conducted between all sample solutions, quality control samples, method blanks, and standards.

Analytical Sequence: The following QC protocol should be employed.

1. Standard S0
2. Standard S1
3. Standard S2
4. Standard S3
5. mid-range ICV(second source)
6. low-level ICV (either source)
7. ICB
8. ICSA
9. ICSAB
10. Sample 1
11. *
12. *
13. *
14. *
15. *
16. *
17. Sample 8
18. CCV
19. CCB
20. Sample 9
21. *
22. *
23. *
24. *
25. Sample 16
26. ICSA
27. ICSAB

- 28. CCV
- 29. CCB

Note: The ICSA and ICSAB sample count as analytical samples between CCV/CCBs.

Any deviations must be approved by the Inorganic Laboratory Manager or the Operations Manager before they can be implemented.

All analyses are documented in the Instrument Run Log (**Attachment 3**) .

9. Data Reduction and Calculations

- 9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Results are reported to two or three significant figures.
- 9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the reporting limit (RL) unless specifically requested by the client.
- 9.3 Soil concentrations are calculated using the equation below:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times Df}{W}$$

Where: C = Concentration in extract (µg/L)
V = Volume of extract (L, 100mL = 0.1L)
Df = Dilution factor (undiluted =1)
W= Dry weight of sample aliquot extracted (g)

- 9.4 Recovery Calculations:

The recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100 \times (SSR-SR)/(SA)$$

Where: SSR = spiked sample result
SR = sample concentration
SA = spike added

- 9.5 Relative Percent Difference Calculations:

The relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100\%$$

$$\text{RPD} = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (replicate)

10. Quality Assurance/Quality Control

- 10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.
- 10.2 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method blank concentration must be less than or equal to reporting limits unless the sample concentration is at least 20 times greater than the blank concentration or less than reporting limits. Corrective action for method blank contamination involves determining the source of the contamination and re-prepping the affected samples in the batch. The analyst may rerun the method blank once as the first step of corrective action.

DoD QSM -Method blank concentrations must be less than or equal to one-half the reporting limit. For common contamination analytes, method blank concentration must be less than or equal the reporting limit.

10.3 Calibration verification --

- 10.3.1 A mid-range ICV analyzed immediately after standards must be within $\pm 10\%$ of the true value. The ICV is an independent source standard purchased from a different vendor than the calibration standards.
- 10.3.1.1 Method 6010C recommends an additional calibration verification using a low-level ICV with a 70-130% criteria, however only the mid-range ICV is required to be from an independent source. In lieu of a second analysis, the response from the S3 standard can be entered back into the curve for verification.
- 10.3.2 The CCV is analyzed a minimum of every 10 samples in the analysis and at the end of the analytical sequence. If the closing CCV does not meet the criteria, the CCV and all analyses from the opening CCV must be re-analyzed after the problem has been eliminated.

- 10.3.3 The ICV and CCV analyses are followed by the ICB and CCB analyses, respectively. The ICV and CCV must pass the $\pm 10\%$ criteria or be re-analyzed.
- 10.4 Use of all standards made from a primary standard must not exceed the primary standard's expiration date.
- 10.5 Matrix spike (MS) samples - A matrix spike is processed with each batch of samples. Spike recoveries must be within 75-125% of the expected value. If the native sample results exceed 4x the spike added, no further action is needed. Note in narrative. Unless superceded by project requirements, it is not necessary to spike Na, K, Ca, and Mg for waters; or Na, K, Ca, Mg, Fe, Al and Mn for soil.

DoD QSM: Spike recoveries must be within 80-120%. Precision requirements are $\leq 20\%$ RPD for both aqueous and soil matrices. There is no corrective action for MS recoveries outside the acceptance range other than data qualification.

- 10.6 Matrix duplicate (DUP) or matrix spike duplicate (MSD) samples (Both options are allowed in method SW6010)- Duplicates are prepped and analyzed with every batch not to exceed 20 samples. Relative Percent Difference (RPD) is calculated for the results of duplicate samples.
- 10.6.1 A limit of 20% RPD shall be used for sample values greater than 5x the MRL.
- 10.6.2 A control limit of (\pm) the MRL level must be used if either the sample or the duplicate value is less than 5x MRL.
- 10.6.3 If one result is above 5x MRL and one below, use the \pm MRL criteria.
- 10.6.4 If both values are below the MRL, no RPD is calculated.

DoD QSM- DUP precision is evaluated for all analytes. Precision requirements are $\leq 20\%$ RPD for both water and soil. There is no corrective action for MS/MD precision outside the acceptance range other than data qualification.

- 10.7 Laboratory Control Sample (LCS) - is prepped with a minimum of every 20 samples of the same matrix.
- 10.7.1 For an aqueous LCS sample, mixed standards are spiked into a beaker of DI water resulting in concentrations approximately $\frac{1}{2}$ the concentration of the high calibration standard and prepped as an aqueous sample. Recoveries must be within the established control limits. The ID of the aqueous LCS sample is LCSW.

10.7.2 For soils, approximately 1g of Teflon Chips (Chemware Ultra-pure PTFE boiling stones, acid washed) used to simulate solid matrix is spiked with standards at the approximate mid-point of the calibration curve. The LCS is then prepped as a soil. The ID for the solid LCS sample is LCSS.

DoD QSM –The acceptance range for both water and soil is 80-120% except silver which has a 75-120% range for soils. When Mitkem in-house LCS limit fall within DoD limit, in-house limits may be reported. If Mitkem in-house limits are wider than DoD limits, the 80-120 % limits must be used.

Laboratory control sample acceptance limits are based on control charts, which are established at Mitkem. These are referred to as in-house limits. Mitkem may also adopt limits from outside agencies when necessary.

10.8 Post digestion spike (PDS) addition - An analyte spike added to a portion of the sample digestate. A PDS is analyzed with each preparation batch of samples (PDS spiking of the same sample as used for matrix spiking is recommended) and is suggested when a new or unusual matrix is encountered within a batch. The acceptance criteria for PDS is 80-120%. The PDS is spiked at the same concentrations as the matrix spike. The PDS concentration should be >10 times the MRL, but < 100 times. If the PDS is not recovered within the specified limits, a matrix effect is confirmed.

DoD QSM: A PDS is required when a new or unusual matrix is encountered within a batch; or when the serial dilution test fails. In addition, a PDS is recommended for projects where all samples are below 50X MDL. The acceptance window is 75-125%.

Corrective action should be taken if the spike concentration is at least 2x the native sample concentration:

- (a) If the MS fails for any analyte but the PDS passes, the sample may be redigested and analyzed, except when low MS recovery can be explained as a historically poor performer (example: Antimony (Sb) for soils) in that matrix. In these instances the analyst may want to discuss the outliers with the project manager or department supervisor. When redigestion is warranted, and the MS fails in the redigestate, a matrix issue is confirmed. If the MS passes for the redigestate, the initial results were due to human error during digestion or spiking.
- (b) If both the MS and PDS fail, the laboratory will perform a dilution test, or may make a reasonable effort to address the matrix interference by performing the method of standard additions (MSA) or internal standard addition.

10.9 Dilution test (Serial Dilution) – should be performed with each preparation batch. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the MDL), an analysis of a 5X dilution should agree within $\pm 10\%$ of the original determination. If not,

a chemical or physical interference effect is confirmed. A serial dilution should be run whenever the PDS fails, to confirm matrix effect.

DoD QSM: A Serial Dilution is required once per preparation batch or when an unusual sample matrix is present. If the SD fails, a PDS must be run.

As a general rule, Mitkem performs a PDS and SD with each preparation batch of samples.

- 10.10 Instrument detection limits (IDLs) are established at the time the instrument is set up and every six months thereafter. Ten solutions of diluted acid (1% HNO₃ and 1.5% HCl) are analyzed on three non-consecutive days. The IDL for a particular element is three times the average standard deviation of the measured concentration for that element. An IDL is valid in the condition that IDL on both instruments are less than MDL currently reported.

DoD QSM: IDLs are established at the time the instrument is set up and whenever significant changes are made

- 10.11 Method detection limits (MDLs) may be established once a year. Mandatory MDL determination has been removed from the SW846 6010C method however until regulatory agencies are in agreement, Mitkem will continue to perform MDL studies. The MDL is obtained by multiplying the standard deviation of seven analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the calculated MDL. An MDL verification check is performed immediately following the MDL study. In lieu of an annual MDL study, the MDL verification can be run quarterly on each instrument. The MDL verification check sample is spiked at approximately 1-4 times the current MDL and prepared as if it were a sample.
- 10.12 Sample Dilutions: When analytes exceed the established linear dynamic range, the sample must be diluted sufficiently to bring those elements into the linear range. The dilution factor (Df) must be integrated into the final concentration calculation.

11. Data Validation and Reporting

- 11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory manager reviews 100% of the data prior to report generation. The QA Director or designee randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

- 11.2 Electronic files are validated by the analyst and transferred to the report generation group via the LIMS. Once data is in LIMS it is reviewed and validated again by the supervisor or his/her designee. The data are then locked and forms may be generated.
- 11.3 Reports are generated by the reporting group in conjunction with the metals lab. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

- 12.1 Corrective action to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.
- 12.2 Corrective action reports (CARs) are initiated through the LIMS in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

- 13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel on the bookcase between the GC/MS and OPrep labs. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan, and have read the Mitkem Contingency Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

- 13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.
- 13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.
- 13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.
- 13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's Quality Assurance Plan.

15. References

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U.S. Environmental Protection Agency. *Inductively Coupled Plasma Atomic Emission Spectroscopy* Method 6010C, SW-846 test methods for evaluating solids waste, Revision 3, Final Update IV, February 2007.

U.S. Environmental Protection Agency. Inductively Coupled Plasma-Atomic Emission Spectrometry Method for the Analysis of Waters and solids, EMMC, July 1992.

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Attachments:

1. **Attachment 1:** Analytes of Interest (Table 1 from SW-846 6010C)
2. **Attachment 2:** DoD QC Requirements, Table B-6,D-18, D-19
3. **Attachment 3:** Standard Preparation Logs
4. **Attachment 4:** Instrument Run Logbook

Attachment 1

Analytes of Interest

TABLE 1

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength ^a (nm)	Estimated IDL ^b (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678 x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227 x2	17
Molybdenum	202.030	5.3
Nickel	231.604 x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO ₂)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980 x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856 x2	1.2

TABLE 1
(continued)

- ^a The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity. Other wavelengths may be substituted (e.g., in the case of an interference) if they provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.
- ^b The estimated instrumental detection limits shown are provided for illustrative purposes only. Each laboratory must determine IDLs and MDLs, as necessary, for their specific application of the method. These IDLs represent radial plasma data and axial plasma IDLs may be lower.
- ^c Highly dependent on operating conditions and plasma position.

Attachment 2:
DoD QC Requirements, Table B-6,D-18, D-19

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18).	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change	Detection limits established shall be \leq MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within \pm 10% of expected value	NA	NA	

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) (ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within $\pm 10\%$ of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within $\pm 10\%$ of expected value GFAA: within $\pm 20\%$ of expected value CVAA: within $\pm 20\%$ of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within $\pm 20\%$ of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch	No analytes detected > ½ RL For common laboratory contaminants, no analytes detected > RL	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	<u>ICS-A:</u> Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) <u>ICS-AB:</u> Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test <i>serial dilution</i>	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	<u>ICP:</u> Perform post-digestion spike (PDS) addition <u>GFAA:</u> Perform recovery test <u>CVAA:</u> Perform matrix spike	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	The spike addition should produce a level between 10 - 100 x MDL.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x MDL	Recovery within 85-115% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard additions (MSA) or internal standard calibration	When matrix interference is suspected	NA	NA	NA	Document use of MSA in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per preparatory batch per matrix	RPD \leq 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

**TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846
METHODS 6010 AND 7470 WATER MATRIX²⁸**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

AG

²⁸ The as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846
METHODS 6010 AND 7471 SOLID MATRIX²⁹

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

5012

²⁹ Some as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

**Attachment 3:
Standard Preparation Logs**

Attachment 4:
Instrument Run Logbook

MITKEM LABORATORIES						SAMPLE RUN LOG: ICAP/4300DV						Date:		Analyst:	
												Spray Chamber: Minicone			
POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		
1			21			41			61			81			
2			22			42			62			82			
3			23			43			63			83			
4			24			44			64			84			
5			25			45			65			85			
6			26			46			66			86			
7			27			47			67			87			
8			28			48			68			88			
9			29			49			69			89			
10			30			50			70			90			
11			31			51			71			91			
12			32			52			72			92			
13			33			53			73			93			
14			34			54			74			94			
15			35			55			75			95			
16			36			56			76			96			
17			37			57			77			97			
18			38			58			78			98			
19			39			59			79			99			
20			40			60			80			100			
Comments:												101			
												102			
												103			

Lot #s

HNO3: _____

STANDARD: _____

HCL: _____

ICV/CCV: _____

CRI: _____

ICSA/ICSAB: _____

MITKEM LABORATORIES						SAMPLE RUN LOG: ICAP/3100XL						Date:		Analyst:	
												Spray Chamber: Minicone			
POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		
1			21			41			61			81			
2			22			42			62			82			
3			23			43			63			83			
4			24			44			64			84			
5			25			45			65			85			
6			26			46			66			86			
7			27			47			67			87			
8			28			48			68			88			
9			29			49			69			89			
10			30			50			70			90			
11			31			51			71			91			
12			32			52			72			92			
13			33			53			73			93			
14			34			54			74			94			
15			35			55			75			95			
16			36			56			76			96			
17			37			57			77			97			
18			38			58			78			98			
19			39			59			79			99			
20			40			60			80			100			
Comments:												101			
												102			
												103			

Lot #s

HNO3: _____

STANDARD: _____

HCL: _____

ICV/CCV: _____

CRI: _____

ICSA/ICSAB: _____

Logbook 00.0126-04/08

Reviewed by: _____

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

For

**Determination of Metals in Water and Soils by SW-846 Method 6020A
Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)**

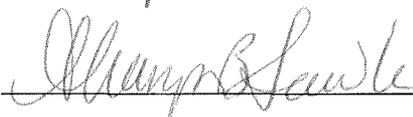
**SOP No.100.0110
Rev. 2**

Signature

Date

QA Director: 

4/22/10

Lab Director: 

4/20/10

Effective Date: 4/29/10

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

For

**Determination of Metals in Water and Soils by SW-846 Method 6020A
Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)**

**SOP No.100.0110
Rev. 2**

1. Scope and Application

This SOP describes the procedures applicable to the analysis of elements listed on page 1 of SW-846 Method 6020A. ICP technology was built upon the same principles used in atomic emission spectrometry. Samples are decomposed to neutral elements in high temperature argon plasma and analyzed based on their mass to charge ratios. An ICP-MS can be thought of as having four main processes, including sample introduction and aerosol generation, ionization by an argon plasma source, mass discrimination, and the detection system.

All matrices, including surface and ground waters, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments and other solid wastes, wipes and air filters are applicable to this analysis. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Total recoverable metals require acid digestion prior to analysis. This SOP pertains to the analysis of digestates and standards.

2. Personnel Qualifications and Responsibilities

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. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. Supervisors/Managers are responsible for ensuring that SOPs are accurate and up to date, and that they are implemented appropriately. Supervisors/Managers review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

- 3.1 Prior to analysis, total (acid-soluble) metals samples must be digested using appropriate sample preparation methods. Dissolved metals do not require digestion. (see **Section 8.1**)
- 3.2 An ICP-MS combines a high-temperature ICP (Inductively Coupled Plasma) source with a mass spectrometer (MS). The ICP source converts the atoms of the elements in the sample to ions. These ions are then separated and detected by the mass spectrometer. For a complete description of ICP-MS see the primer at <http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm>, this gives a concise breakdown of how an ICP/MS works.

4. **Sample Preservation, Containers, Handling, and Storage**

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the client. For metals analysis, water samples are collected in 1 liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.
- 4.2 Sample holding time for metals analysis is 180 days from VTSR for both water and soil for all metals other than mercury.

5. **Interferences and Potential Problems**

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICP-MS

- 5.1 Isobaric elemental interferences are caused by isotopes of different elements, which form singly or doubly charged ions of the same nominal mass-to-charge ratio.
- 5.2 Abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small m/z peak is being measured adjacent to a large one (also called wing overlap).
- 5.3 Isobaric Polyatomic Ion Interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use.
- 5.4 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 salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the

nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

- 5.5 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.
- 5.6 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 or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.
- 5.7 Contamination by samples: Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates, and other samples containing high concentrations of inorganic substances are processed and analyzed.
- 5.8 Contamination by indirect contact: It is imperative that every piece of the apparatus that is directly or indirectly used in the collection, processing, and analysis of ambient water samples be cleaned.
- 5.9 Contamination by airborne particulate matter: Samples may be contaminated by airborne dust, dirt, particles, or vapors from unfiltered air supplies; nearby corroded or rusted pipes, wires, or other fixtures; or metal-containing paint.
- 5.10 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. Mitkem performs the following analyses in each analytical sequence.
 - a) Serial Dilution Test: One sample per matrix per batch; A 5x dilution must agree to within 10% of the original determination if the analyte concentration is sufficiently high (minimally a factor of 10 times the PQL of the sample). If not, a chemical or physical interference effect is suspected. Samples identified as field blanks or PEs should not be used for serial dilutions.
 - b) Post Digestion Spike Addition: If the spike (pre-digestion) recovery of an analyte falls outside of the control limits, a portion of the same sample (digestate) is spiked with a post-digestion spike. The PDS should be recovered to within 80-120% of its true value. The spike addition should produce a minimum level of 10 x and a maximum of 100x the PQL. If both the Matrix spike and the PDS fail, matrix effects are confirmed.

6. Equipment and Apparatus

6.1 Glassware

6.1.1 Class A volumetric flasks:

- 100 mL and 50 mL

6.1.2 Class A volumetric pipettes:

- 10-100 μ L adjustable Eppendorf.
- 100-1000 μ L adjustable Eppendorf.
- 1-5mL adjustable Fisher Scientific.

6.1.3 15mL Evergreen Polystyrene ICP tubes.

6.1.4 100 μ L and 1mL fixed Wheaton.

6.2 ICP-MS- Thermo Electron Xseries2 (Instrument ID: X1)

The built-in radio-frequency generator is FCC compliant.

The high purity grade (99.9%) argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.2.1 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Method detection limits, precision and interference effects must be investigated and established for each analyte. All measurements must be in the instrument operational range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

6.2.2 Instrument Maintenance: See manufacturer's operation manual for additional information.

6.2.2.1 Peristaltic pump tubing should be replaced after approximately 20 hours of instrument run time. Relative standard deviations within samples containing measurable analytes that are above 3% indicate excessive tubing wear. Record routine maintenance in the instrument run logbook.

6.2.2.2 Torch and cones should be inspected daily for obvious buildup. Degraded performance will be clear when the daily performance check is run and fails or barely passes. Problems may be rectified by adjusting the instrument settings in a tune, and this should be attempted before cleaning the system. Light elements (Be) will be the first to be impacted by a dirty sample introduction system or cones.

6.2.2.3 The daily performance check will also indicate the need for mass calibration if responses are acceptable and mass verification differs by more than 0.1 from the true value

6.2.3 Troubleshooting: See manufacturer's operation manual.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors. All solutions are traceable and meet with Mitkem's high purity requirements. Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. ***Please note that standards and reagents from other vendors than listed below could be used as long as the standards are of high purity (>96%) and traceable to reference materials.*** All standards are labeled as instructed in Mitkem SOP# 80.0001 Standard Preparation, Equivalency and Traceability. Reagents are labeled as instructed in Mitkem SOP# 80.0013 Reagent Purchasing and Tracking.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade.

7.2 Nitric acid (conc.), HNO₃, Trace Grade.

7.3 Reagent water. Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. It is also referred to as DI Water.

7.4 Primary Standards: Mixed calibration standards and individual analyte standards; obtained from High Purity Standards, come in several stock solutions.

7.4.1 Intermediate (Secondary) and Working Standards: Dilutions of the above primary standards in acidified reagent water as appropriate.

7.4.1.1 A high level working calibration standard containing all elements required for calibration is prepared from the primary standards. This standard ranges in concentration from 0.2 to 50 ppm and is used as the upper level of the curve (S6). From this standard the other calibration standards are prepared (S5 to S1) by dilution.

S5=1:2x dilution of S6
S4=1:10x dilution of S6
S3=1:40x dilution of S6
S2=1:100x dilution of S6
S1=1:200x dilution of S6

See **Table 2** for individual element concentrations.

- 7.4.2 Working Calibration standards and QC solutions are made up on a daily basis. All standards and solutions are documented in the lab's standard logbooks. Each laboratory area has Primary, Intermediate and the Working Standard Logbooks in hardcopy or in LIMS. Concentrations of working standards and the associated QC limits can be found in LIMS within the individual testcode SPECs, as identified by Standard or QC Sample name (i.e.: ICAL1) or refer to **Table 1**.
- 7.5 Tuning Solution and Mass Calibration: Prepare a dilution of mixed calibration standards with Beryllium, Magnesium, Cobalt, Indium and Lead at 10ug/L. The solution should be in 1%HNO₃.
- 7.6 Initial Calibration Verification Standard (ICV): An independent source standard prepared at a concentration near the high end of the calibration curve. This standard may also be purchased. See **Table 1**.
- 7.7 Low Level Initial Calibration Verification Standard (LLICV): A custom standard at or slightly below the report limit.
- 7.8 Interference Check Standard (ICS): QC Standards ICESA and ICESAB stock solutions are obtained from Accustandard. Prepare ICESA and ICESAB fresh daily. See **Table 1**.
- 7.9 Matrix Spike and Laboratory Control Sample: See preparatory methods for vendor information. LIMS will have final concentrations expected in the digestates in the LIMS testcode SPECs. See **Table 1**.

8. Procedure

- 8.1 The methods used for sample digestion or preparation are as follows:
- 8.1.1 For water samples to be analyzed for total acid soluble/recoverable metals determination: a 50mL aliquot of the unfiltered (well-shaken) sample is digested with nitric and hydrochloric acids using Mitkem **SOP #100.0003**, Methods SW-846 3005/3010.

- 8.1.2 For water samples to be analyzed for dissolved metals determination: a 50mL aliquot of the filtered (0.45 μ m filter) sample is nitric acid preserved using Mitkem **SOP #100.0003**, Methods SW-846 3005/3010. No digestion is required.
- 8.1.3 For soil samples to be analyzed for total metals determination: a representative sample (between 1.0 and 2.0 gram) is digested with nitric acid using Mitkem **SOP #100.0104**, Method SW-846 3050.
- 8.2 Precalibration routine
- 8.2.1 Prior to calibration, the instrument must be allowed to become stable. This may take up to a half-hour. Conduct any necessary mass calibration and resolution routines to bring peak width within the manufacturer's specifications and adjust mass calibration to within 0.1u over the range of 6 to 210u. The resolution must be verified to be less than 0.9amu full width at 10% peak height.
- 8.2.2 Performance Check: Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least five integrations. The %RSD of the absolute signals for all of the multiple integrations in the tuning solution (as calculated by the instrument) must be $\leq 5.0\%$ for each analyte.
- 8.2.3 Internal Standardization: For full range mass scans, a minimum of 5 internal standards (IS) must be used. The IS chosen must be consistent throughout the entire run sequence. IS shall be present in all samples, standards and blanks at the same levels. No IS are used in the tuning solution. If dilutions are performed on the digested samples, then the IS must be added after the dilution. IS are added by the instrument. The IS used at Mitkem are listed in Table 3.
- 8.3 Calibration: Instrument calibration is required each time the instrument is set up for a sequence or after a continuing calibration failure. Extensive instrument maintenance or significant analytical changes would require re-calibration as well.
- 8.3.1 Calibrate the instrument with one blank standard and at least 3 non-zero standards. See **Table 2**. One standard must be at, or below, the PQL. With a multi-point curve, the upper quantitation limit (UQL) may exceed the highest concentration calibration point and can be defined as the "linear dynamic" range. Alternately, the calibration curve could be prepared daily with a minimum of a calibration blank and a single standard at the appropriate concentration to effectively outline the desired quantitation range. The multi-point curve is the preferred method of calibration at Mitkem.
- 8.3.2 A minimum of 3 replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

- 8.3.3 The calibration curve shall be calculated using linear regression by plotting the concentration of the standard in ug/L on the X-axis versus the corrected instrument response on the Y-axis. The correlation coefficient, r , for the calibration curve must be ≥ 0.998 .

DoD requires a correlation coefficient $r \geq 0.995$

- 8.3.3.1 If the recommended linear response cannot be attained using a minimum of three non-zero calibration standards, consideration should be given to adding more standards, particularly at the lower concentrations, in order to better define the linear range and the lower limit of quantitation. Conversely, the extreme upper and lower calibration points may be removed from the multi-point curve as long as three non-zero points remain such that the linear range is narrowed and the non-linear upper and/or lower portions are removed.

8.4 Calibration Verification

- 8.4.1 Initial Calibration Verification (ICV): After successful initial calibration, the accuracy of the curve is verified for every analyte by the analysis of the second or independent source ICV for each mass used to report final data results. The concentration of the ICV is near the upper end of the calibration range. Recovery should be within 90-110% recovery to verify the curve.
- 8.4.2 Low Level Initial Calibration Verification (LLICV): After successful ICV analysis, it is recommended that the accuracy of the low-end of the calibration range be verified by the analysis of a LLICV for each mass used to report final data results. The standard is prepared from individual elements standards. The recovery should be within $\pm 30\%$. Alternatively, the low standard (S1) of a multi-level curve could be plugged back into the curve for verification.

DoD requires the LLICV meet 80-120 % recovery criteria when performing a one point calibration.

- 8.4.3 Continuing Calibration Verification (CCV): To ensure calibration accuracy during each analytical sequence, a CCV shall be analyzed and reported for each mass used for reporting final results for each element, at a frequency not to exceed every 10 samples during an analytical sequence. The CCV must be analyzed at the beginning of the run and after the last analytical sample. The recovery should be within 90-110% recovery to accept ongoing calibration accuracy. The analytical sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCVs meet the previously mentioned criteria.

- 8.4.3.1 CCV standards are prepared from the same source as the calibration and are at the mid-level (S5) concentration used during initial calibration.
 - 8.4.3.2 The same CCV is used throughout the entire analytical sequence.
 - 8.4.3.3 The CCV is prepared in the same acid matrix as the calibration standards.
- 8.5 Initial and Continuing Calibration Blanks (ICB/CCB): The ICB and CCB are identical in composition to the calibration blank (S0) used in the initial calibration. The ICB/CCB are analyzed immediately after the ICV/CCV to monitor for potential carryover of analytes. If the absolute value of the ICB/CCB result exceeds the PQL, the analysis is stopped and the problem corrected. The instrument must then be recalibrated.
- 8.6 Sample Analysis
- 8.6.1 Interference Check Sample (ICS): To verify corrections for elemental and polyatomic isobaric interferences and to monitor for all interferences, an ICS is analyzed and reported for all elements on the Target Analyte List (TAL) immediately after the initial calibration but not before the ICV/ICB pair. The analysis of the ICS is immediately followed by the analysis of the CCV/CCB pair. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferences and Solution AB consists of the analytes mixed with the interferences. The ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.
 - 8.6.2 Matrix Spike Sample (MS): Spiked samples are analyzed to provide information about the effect of sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion reagents. At least one spike sample is performed on each batch of samples of a similar sample matrix type (water, soil...). Samples identified as field blanks or PEs should not be used for matrix spike samples. In some cases the sample requested for use as a matrix spike will be identified. The MS is spiked with the same solution as is used for the LCS, at the same concentration. Percent recovery is calculated for each analyte added.
 - 8.6.3 Duplicate Sample (DUP): Duplicate samples are analyzed to provide information about the precision of the methodology for each element. Samples identified as field blanks or PE should not be used as duplicates. In some cases the sample requested for use as a duplicate will be identified. Relative Percent Difference is calculated for each analyte detected in the duplicate samples.
 - 8.6.4 Laboratory Control Sample (LCS): The LCS is prepared by spiking an aliquot of reagent water or digesting an aliquot of an SRM. One LCS is prepared per

preparation batch of water or soil samples in a batch. Percent Recovery is calculated for each analyte in the LCS. The LCS must include all elements of interest.

- 8.6.5 After completion of the initial requirements of this method, field samples should be analyzed in the same operational manner used in the calibration routine. A rinse blank is run between all sample solutions, quality control samples, method blanks, and check solutions. Samples that exceed the linear dynamic range must be diluted and reanalyzed. Dilution factors should be appropriate to bring the readings within the upper 75% limit of the linear range.

Analytical Sequence:

1. Performance Check
2. Standard S0 (Blank std)
3. Standard S1 (Low std)
4. Standard S2
5. Standard S3
6. Standard S4
7. Standard S5 (Mid-range std =CCV)
8. Standard S6 (High std)
9. ICV
10. ICB
11. LLICV
12. ICSA
13. ICSAB
14. CCV
15. CCB
16. Samples
17. CCV (not to exceed 10 samples since last CCV)
18. CCB
19. Samples
20. CCV(not to exceed 10 samples since last CCV)
21. CCB

Note: If the last CCV, CCB meet the QC criteria the analytical run can be continued with using the same sequence protocol outlined above.

All analyses are documented in the ICP/MS Instrument Run Log.

9. Data Reduction and Calculations

- 9.1 Raw data can be evaluated on-screen using the PlasmaLab software package. PlasmaLab will identify all non-conforming data points and provide the analyst with details as to what elements or masses are meeting or not meeting pre-set quality control limits. Any data point

can be chosen using the cursor and right clicked to enable the *tooltip* reading pane on the bottom of the screen. The *tooltip* screen shows additional information including the type of quantitation or detector mode used for the mass calibration and data result. Data that are underlined also have been flagged to alert the analyst to the following (more information may be found in the Thermo Electron Xseries2 operation manual):

I=Invalid Calibration; alerts the analyst that there was a QC failure during the calibration and is common with certain alternate masses not used for quantitation.

T=Tripped; alerts the analyst that the detector was switched from pulse to analog as is done with high concentration analyses.

D= Semi-quantitative calibration mode used instead of Fully-; result is an estimate due to calibration issues as in "T".

M=Maximum exceeded; result is above the upper calibration range however not necessarily the linear range.

9.2 After completion of the analyses, the data files are exported to the server (\CLP Files directory). Data may be printed at this point or saved to pdf print file.

9.3 Soil samples are reported on a dry weight basis.

9.3.1 Percent Solids are calculated using the following formula:

$$\% \text{ solids} = \frac{\text{DW} \times 100}{\text{WW}}$$

DW = Sample weight (g) dried

WW = Sample weight (g) before drying

10. Quality Assurance/Quality Control

10.1 Instrument detection limits (IDLs) are a useful tool to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the PQL, nor should they be used in establishing this limit. IDLs are established initially when the instrument is set up and then on a quarterly basis. IDL values are uploaded to the LIMS system for each instrument.

IDLs in $\mu\text{g/L}$ can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). It may be helpful to compare the calculated IDLs to the testcode PQLs. It should be understood that the IDL must be less than the PQL. The PQL needs to be verified initially by analysis of a lower

limit of quantitation check (LLQC) sample and on an as needed basis to verify the lab's ability to detect elements at the reporting level. See **Section 10.4**.

DoD recommends performing an IDL study at instrument set-up or after significant maintenance or changes. The IDL \leq LOD.

- 10.2 The linear dynamic range (LDR) is established when the system is first setup, or whenever significant instrument components have been replaced or repaired, and on an as needed basis only after the system has been successfully calibrated using either the single or multi-point standard calibration approach. The upper limit of the linear dynamic range needs to be established for each wavelength utilized by determining the signal responses from a minimum of three, preferably five, different concentration standards across the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

A standard at the upper limit should be prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within $\pm 10\%$ of the true value. New upper range limits should be determined whenever there is a significant change in instrument response. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply a spectral correction, the correction might not be valid and those analytes where the spectral correction has been applied may be inaccurately reported.

DoD requires LDR or upper limit check standard every six months, at a minimum.

- 10.3 Method detection limits (MDLs) are established annually however there is *no requirement* in the current SW-846 6020A method.

DoD requires an MDL study when the instrument is initially set up and quarterly LOD verifications thereafter. See Mitkem SOP #80.0005 for more details.

- 10.4 The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits (PQL) and on an as needed basis to demonstrate the desired detection capability. This check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. PQLs are verified when all analytes in the LLQC sample are detected within $\pm 30\%$ of their true value.

- 10.5 Initial Precision and Accuracy (IPANDA studies): Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. The precision and accuracy studies are done as a combination of a preparative method and analysis. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is

achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made.

- 10.6 Performance Check: If the mass calibration is not within the 0.1u over the range of 6 to 210 u, or the %RSD of all the integrations of the absolute signals of the analytes exceeds 5.0%, the analysis is terminated and the problem corrected. The instrument would then be re-tuned before any further calibration.
- 10.7 Initial Calibration Verification (ICV): The measurements should be within 90-110% recovery to verify the curve. If the ICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated before reanalyzing the ICV.
- 10.8 Low Level Initial Calibration Verification (LLICV): The measurements should be within 70-130% recovery to verify detection at the low end of the curve. If the LLICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated.
- 10.9 Continuing Calibration Verification (CCV): If the deviation of the CCV is greater than the control limits of 90 - 110% recovery, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the elements affected.
- 10.10 Blanks: There are three types of blanks required by this method. The calibration blank (S0) is used in establishing the initial calibration. This same blank is used for the initial and continuing blanks (ICB/CCB) to monitor for carryover. The Method blank, or Preparation blank, is used to monitor contamination from preparation and analysis. A Rinse blank is used to flush the system between standards and samples.
 - 10.10.1 ICB/CCB: If the absolute value of an analyte result exceeds the PQL the analysis is terminated and the problem corrected. The instrument would then require recalibration and reanalysis of all samples that were analyzed since the last compliant calibration blank. Calibration Blanks consist of the same concentrations of the same acids used to prepare the final dilution of the calibrating solutions. We use 1% (v:v) Nitric acid and 0.5% (v:v) HCl in reagent water. See section 7.2 of SW-846 6020A.
 - 10.10.2 Method/Preparation Blanks (MB): The MB contains all the reagents in the same volumes as the samples it is prepared with. The MB is carried through the full procedure and has the same acid concentration in the final digestate as the associated samples. Analyze the MB in the same manner as a sample. Each preparation batch of 20 or less field samples has a minimum of one MB. For positive MB concentrations (the MB may be rerun once to verify results):

- 10.10.2.1 If the absolute value of the concentration is less than or equal to the PQL in the MB, no corrective action is needed.
- 10.10.2.2 If the analyte concentration is above the PQL in the MB, the lowest concentration of that analyte in the associated batch samples (not field blanks) is reportable if it is greater than or equal to 10 times the blank concentration. Otherwise all associated batch samples, with the analyte concentration less than 10 times the blank concentration and above the PQL, will be redigested and reanalyzed with new QC for that analyte. No blank correction is performed.

DoD requires no analyte in the MB detected $> \frac{1}{2}$ PQL or greater than 1/10 the amount measured in any associated sample. Common lab contaminants should be $<$ PQL.

- 10.10.2.3 If the analyte concentration in the sample(s) is below the PQL, the samples may be reported despite the method blank contamination. This should be noted in the narrative.

10.10.3 Rinse Blanks consist of 2% HNO₃ and 0.5% HCl in reagent water.

10.11 Interference Check Sample (ICS):

- 10.11.1 Analytical results of Solution A (ICSA) shall fall within the control limits of ± 2 times the PQL or $\pm 20\%$ of the analyte's true value (the true value shall be zero unless otherwise stated or determined by multiple analyses as directed in section 9.7 of method 6020A) in the ICSA, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

DoD requires the absolute value of concentration for all non-spiked elements $<$ LOD, unless they are a verified trace impurity from one of the spiked elements.

- 10.11.2 Analytical results of Solution AB (ICSAB) shall fall within the control limits of ± 2 times the PQL or $\pm 20\%$ of the analyte's true value for the analytes included in the ICSAB, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

DoD requires recoveries within $\pm 20\%$ of the analyte's true value for those analytes included in the ICSAB.

- 10.12 Matrix Spike analysis is acceptable if recovery is within $\pm 25\%$. If recovery is outside of this limit for any analyte then the associated samples are flagged. The only exception to these criteria is when the sample concentration exceeds the concentration of the spike added by a

factor of four or more. In this case no flagging is applied. When recoveries fail to meet criteria, a post digestion spike (PDS) is required.

DoD recovery limits for MS analyses are the same as LCS limits. DoD QSM does not give a set of limits for method SW6020, therefore, method SW6010 LCS limits or 80-120% will be used (silver in soil is 75-120%).

- 10.13 Post digestion spike (PDS): When matrix spike recovery fails, a post digestion spike is performed on the same sample as the spike was done originally. Spike the unspiked aliquot of the undiluted digestate to produce a minimum level of 10 x PQL and a maximum of 100x PQL. PDS recoveries outside of $\pm 20\%$ of the true value confirm matrix interference and the associated samples are flagged.

DoD allows PDS recoveries up to $\pm 25\%$.

- 10.14 Duplicate: A control limit of $\pm 20\%$ for RPD is used for original and duplicate samples. Corrective action required as a result of the duplicate analyses would be to analyze a serial dilution on the sample.
- 10.15 Serial Dilution (SD): If the analyte concentration is sufficiently high (minimally, a factor of 10 times above the PQL), a 5 times dilution should agree to within $\pm 10\%$ of the original sample analyte determination. If not, a chemical or physical interference effect is suspected and the associated samples are flagged.
- 10.16 Laboratory Control Sample (LCS): Recovery is calculated for all analytes and is to be within the control limits of $\pm 20\%$. In the event that the LCS does not meet criteria it may be reanalyzed once. If still unacceptable, associated samples must be redigested. Solid Reference Material (SRM) may be used as an LCS for soil sample batches. The manufacturer's established PT PAL acceptance criteria should be used instead of the $\pm 20\%$ criteria.
- 10.17 Internal Standards (IS): Internal standard response is monitored throughout the analytical sequence. Ratios of the raw uncorrected IS responses between isotopes should also be monitored routinely. This information can be useful for correcting problems caused by mass dependent drift, errors incurred in the IS addition, or background contributions from samples that cause high bias. If the intensity of any internal standard in a sample falls below 70% of the intensity of that internal standard in the initial calibration standard S0, a significant matrix effect must be suspected. Under these conditions, the established PQL has degraded and the correction ability of the internal standard technique becomes questionable. Make sure the instrument has not drifted by observing the internal standard intensities in the nearest clean matrix. If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold and reanalyzed with the

addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit.

DoD Internal standard intensity limits are 30-120% of the IS in the initial calibration.
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11. Data Validation and Reporting

- 11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks and instrument logs are reviewed and signed regularly by the supervisor. The laboratory supervisor or another senior chemist reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.
- 11.2 The reporting group generates reports. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

- 12.1 Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.
- 12.2 Corrective action reports (CARs) are initiated in the event of an out of control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

- 13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. These sheets are stored in the bookcase adjacent to the Organic Analysis lab. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Manual. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.
- 13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.
- 13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

- 13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.
- 13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Method 6020A, Inductively Coupled Plasma- Mass Spectrometry. Revision 1, February 2007, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1 April 2009.

Operation manual(s) for Thermo Electron Xseries2

<http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm>, internet reference for how an ICP/MS works.

Attachments:

Table 1: QC/ Standard Concentrations

Table 2: Calibration Standard Concentrations

Table 3: Internal Standards

Attachment 1: DoD Table F-8

Table 1: QC/ Standard Concentrations

ELEMENT	MASS	PPB 6020 LLICV	PPB 6020 ICV	PPB 6020 CCV	PPB 6020 ICSA	PPB 6020 ICSAB	PPB 6020 LCS	PPB 6020 MS
Be	9	1	50	100	X	X	50	50
B *	10	50	1000	1000	X	X	1000	1000
Na	23	250	15000	25000	250000	250000	5000	5000
Mg	24	250	10000	25000	100000	100000	5000	5000
Mg	25	250	10000	25000	100000	100000	5000	5000
Mg	26	250	10000	25000	100000	100000	5000	5000
Al	27	20	2000	2000	100000	100000	2000	2000
K	39	250	50000	25000	100000	100000	5000	5000
Ca	44	250	5000	25000	300000	300000	5000	5000
V	51	52	500	100		200	500	500
Cr	52	2	200	100		200	200	200
Cr	53	2	200	100		200	200	200
Fe	54	100	1000	10000	250000	250000	1000	1000
Mn	55	15	500	100		200	500	500
Fe	56	100	1000	10000	250000	250000	1000	1000
Fe	57	100	1000	10000	250000	250000	1000	1000
Co	59	1	500	100		200	500	500
Ni	60	1	500	100		200	500	500
Ni	61	1	500	100		200	500	500
	63	2	250	100		200	250	250
Cu	65	2	250	100		200	250	250
Zn	66	5	500	100		100	500	500
Zn	67	25	500	100		100	500	500
Zn	68	25	500	100		100	500	500
As	75	2	400	100		100	40	40
Se	78	5	500	100		100	50	50
Se	82	5	500	100		100	50	50
Mo *	95	2	250	100	2000	2000	100	100
Mo *	97	2	250	100	2000	2000	100	100
Ag	107	1	50	100		50	50	50
Ag	109	1	50	100		50	50	50
Cd	111	1	50	100		100	50	50
Cd	114	1	50	100		100	50	50
Sb	121	2	100	100	X	X	100	100
Sb	123	2	100	100	X	X	100	100
Ba	135	10	2000	1000	X	X	2000	2000

Ba	137	10	2000	1000	X	X	2000	2000
Tl	203	1	500	100	X	X	50	50
Tl	205	1	500	100	X	X	50	50
Pb	206-208	1	500	100	X	X	20	20

Bold are Reported Mass

X= no requirement for 6020A

*B and Mo not listed in
6020A

Table 2: Calibration Standard Concentrations

ELEMENT	MASS	ppb	ppb	ppb	ppb	ppb	ppb	ppb
		S1	S2	S3	S4	S5	S6	PQL
Be	9	1	2	5	20	100	200	1
B	10			50	200	1000	2000	50
Na	23	250	500	1250	5000	25000	50000	500
Mg	24	250	500	1250	5000	25000	50000	500
Mg	25	250	500	1250	5000	25000	50000	500
Mg	26	250	500	1250	5000	25000	50000	500
Al	27	20	40	100	400	2000	4000	20
K	39	250	500	1250	5000	25000	50000	500
Ca	44	250	500	1250	5000	25000	50000	500
V	51		2	5	20	100	200	5
Cr	52		2	5	20	100	200	2
Cr	53		2	5	20	100	200	2
Fe	54	100	200	500	2000	10000	20000	200
Mn	55	1	2	5	20	100	200	5
Fe	56	100	200	500	2000	10000	20000	200
Fe	57	100	200	500	2000	10000	20000	200
Co	59	1	2	5	20	100	200	1
Ni	60	1	2	5	20	100	200	1
Ni	61	1	2	5	20	100	200	1
Cu	63		2	5	20	100	200	5
Cu	65		2	5	20	100	200	5
Zn	66		2	5	20	100	200	5
Zn	67		2	5	20	100	200	5
Zn	68		2	5	20	100	200	5
As	75	1	2	5	20	100	200	2
Se	78			5	20	100	200	5
Se	82			5	20	100	200	5
Mo	95	1	2	5	20	100	200	2
Mo	97	1	2	5	20	100	200	2
Ag	107	1	2	5	20	100	200	1
Ag	109	1	2	5	20	100	200	1
Cd	111	1	2	5	20	100	200	1
Cd	114	1	2	5	20	100	200	1
Sb	121		2	5	20	100	200	2
Sb	123		2	5	20	100	200	2
Ba	135	10	20	50	200	1000	2000	10
Ba	137	10	20	50	200	1000	2000	10
Tl	203	1	2	5	20	100	200	1
Tl	205	1	2	5	20	100	200	1
Pb	206-208	1	2	5	20	100	200	1

Table 3: Internal Standards

Concentrations chosen to yield approximately
700,000 raw counts

element	ppb
Li6	100
Sc	50
Y	15
Rh	20
In	15
Tb	15
Ho	15
Lu	10
Bi	15

**Attachment 1:
DoD Table F-8**

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be \leq LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Tuning	Prior to ICAL.	Mass calibration \leq 0.1 amu from the true value; Resolution $<$ 0.9 amu full width at 10% peak height; For stability, RSD \leq 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) for all analytes (minimum one high standard and a calibration blank)	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within $\pm 10\%$ of true value.	Verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All analytes within $\pm 10\%$ of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within $\pm 20\%$ of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Linear dynamic range or high-level check standard	Every 6 months.	Within $\pm 10\%$ of true value.	NA.	NA.	
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and greater than $\frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $> RL$ (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prepare and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run and every 12 hours.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within $\pm 20\%$ of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests (dilution test and post-digestion spike addition) are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD < 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table F-8. Trace Metals Analysis by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) (Method 6020) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution test	One per preparatory batch.	Five-fold dilution must agree within $\pm 10\%$ of the original measurement.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations $> 50 \times$ LOQ.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples $< 50 \times$ LOD.	Recovery within 75-125% (see Table B-1).	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 \times LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Internal standards (IS)	Every sample.	IS intensity within 30-120% of intensity of the IS in the ICAL.	Reanalyze sample at 5-fold dilution with addition of appropriate amounts of internal standards.	Flagging criteria are not appropriate.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

**MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.**

STANDARD OPERATING PROCEDURES

for

**Mercury Analysis in Aqueous and Soil Samples
by Flow Injection Mercury System (FIMS)
for Cold Vapor Atomic Analysis**

by

SW846 Method 7470A/7471B

Rev. 10

Signature

Date

QA Director:  6/15/10

Lab Director:  6/15/10

Effective Date: 6/22/10

**MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.**

STANDARD OPERATING PROCEDURE

for

**Mercury Analysis in Aqueous and Soil Samples
by Flow Injection Analysis System
for Cold Vapor Atomic Analysis**

by

SW846 Method 7470A and 7471B

Rev. 10

1. Scope and Application

This SOP describes the procedures applicable to the preparation and analysis of mercury in aqueous and soil samples. Matrices include ground waters, aqueous samples, soils, sediments, sludges, and TCLP. All samples require digestion prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 The aqueous samples are digested with concentrated HNO₃, concentrated H₂SO₄, potassium permanganate and potassium persulfate at 95°C. The procedure converts various organically bound compounds and inorganic forms

of mercury into mercuric ions, which can be analyzed with a Flow Injection Analysis System (FIAS) for atomic spectroscopy. The soil/sediment samples are digested using aqua regia and potassium permanganate at $95\pm 3^\circ\text{C}$ and analyzed same as the aqueous samples.

- 3.2 The mercury ions formed during the digestion step are reduced to the elemental state and aerated into an absorption cell. Absorbance is measured at 253.7nm and is a function of mercury concentration.

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For mercury analysis by SW 846 method 7470A water samples are collected in one-liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

- 4.2 All samples are stored at room temperature until analyzed.

- 4.3 Sample hold time for mercury analysis by SW 846 methods 7470A and 7471B is 28 days from date of sample collection.

5. Interferences and Potential Problems

- 5.1 Sulfides at levels above 20 mg/L or 20mg/Kg interfere. Potassium permanganate is added to the samples to eliminate possible interference of sulfides.

- 5.2 Copper at levels above 10 mg/L or 10mg/Kg interfere.

- 5.3 Seawaters, brines and industrial effluents high in chlorides interfere and require additional potassium permanganate for conversion to free chlorine. Free chlorine also absorbs radiation at 253.7nm. Therefore, the free chlorine is removed by addition of hydroxylamine sulfate reagent. In addition, the dead air space in the BOD bottle must be purged before adding the stannous sulfate.

- 5.4 Some volatile organic materials absorb at 253.7nm and may interfere.

6. Equipment and Apparatus

Equipment and instrumentation used in this analysis method include:

- 6.1 Equipment:

- 6.1.1 Perkin-Elmer FIMS 100.
- 6.1.2 Printer.
- 6.1.3 Wheaton BOD bottles.
- 6.1.4 Top loading balance - capable of accurate measurement to 0.01gram.
- 6.1.5 Hot Plate with graphite block digester

6.2 Preventative Maintenance:

- 6.2.1 Pump tubing is replaced every 48 hours of instrument run time.
- 6.2.2 The windows of the optical cell are cleaned whenever the cell is replaced.
- 6.2.3 The inside of the optical cell is cleaned once every 48 hours of instrument run time.

6.3 Troubleshooting - Refer to the FIMS Analysis manual.

6.4 Glassware

- 6.4.1 100mL Class "A" volumetric flasks.
- 6.4.2 Class "A" volumetric pipettes ranging from 10 μ L to 1.0mL.
- 6.4.3 100 and 250mL Class "A" graduated cylinder or equivalent.

7. **Reagents and Standards**

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.

7.1 Sulfuric Acid: Ultra Trace Grade

7.2 Nitric Acid: Ultra Trace Grade

- 7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.
- 7.4 Stannous chloride solution:
15 g SnCl_2 to 1000mLs of 3% HCl solution.
- 7.5 Sodium chloride-hydroxylamine sulfate solution:
12 g NaCl and 12g hydroxylamine sulfate to 100mLs of reagent water.
- 7.6 5% Potassium permanganate solution:
Dissolve 50g of KMnO_4 in 1000mLs of DI H_2O .
- 7.7 5% Potassium persulfate solution:
Dissolve 50g of $\text{K}_2\text{S}_2\text{O}_8$ in 1000mL of DI H_2O .
- 7.8 Stock mercury solution(Primary):
1000 mg/L purchased commercially.
- 7.9 Stock mercury solution(Independent Source):
1000 mg/L purchased commercially.
- 7.10 Mercury standards are stored in Wet Chem cupboards. Expiration dates for the standards are either the date one year from date of receipt or that designated by the manufacturer whichever comes first. Reagents and intermediate standards have a 28-day expiration period.
- 7.11 3% HCl: 30mL concentrated HCl diluted to 1L with DI H_2O .
- 7.12 HCl: conc., Ultra Trace grade.
- 7.13 Aqua Regia: prepare immediately before use by carefully adding three volumes of conc. HCl to one volume HNO_3 .

8. Procedure

8.1 Preparation

- 8.1.1 Aqueous Samples: Transfer 100mL of sample or an aliquot diluted to 100mL to a reagent water-rinsed BOD bottle with a graduated cylinder. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3 , mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate

solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool samples and add 6mL sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION:** Do this addition under the hood, Cl₂ could be evolved. Record all volumes and reagents in the Mercury Digestion logbook, (**Attachment 1**). Immediately prepare for analysis.

- 8.1.2 Soil Samples: Weigh a 0.50 – 0.60g portion of a well homogenized untreated sample and place in the bottom of a BOD bottle. See SOP 110.0039 for sub-sampling techniques. Add 5mL of reagent water and 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50mL reagent water and 15mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool and add 6mL of the sodium chloride – hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION:** Do this addition under the hood, Cl₂ could be evolved. Add reagent water to a final volume of 100mL. Pour an aliquot into a polyethylene tube for analysis. Be careful to avoid pouring the sediment into the tube. Record all volumes and reagents in the Mercury Digestion logbook.

8.2. Calibration Standards

- 8.2.1 Working Calibration standards are prepared from a mercury intermediate standard at 500µg/L. The intermediate is prepared by pipetting 50µL of 1000mg/L Primary stock standard into a 100mL volumetric flask. Bring up to volume with 3% HCl.

Label standard as Ilyymmdd#, where:

I I = Inorganic Intermediate

yy = year of preparation

mm = month of preparation

dd = day of preparation and

= Sequential letter of intermediate standards prepped on this day.

All standard preparation information is documented in the Metals Standard Receipt/Preparation Logbooks (Primary, Intermediate and Working).

Into BOD bottles containing approximately 10mL DI H₂O, pipet the following volumes of intermediate standard in order to achieve the corresponding final working standard concentrations:

<u>Volume</u>	<u>Concentration ($\mu\text{g/L Hg}$)</u>	<u>Standard ID</u>
40 μL	0.2	S1
200 μL	1.0	S2
400 μL	2.0	S3
1mL	5.0	S4
2mL	10.0	S5
0mL	0.0	S0

8.2.2 Depending on the matrix of samples being prepared, follow the digestion as follows:

8.2.2.1 Soil Calibration standards, add 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at $95\pm 3^{\circ}\text{C}$. Cool; then add 50 mL reagent water and 15 mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at $95\pm 3^{\circ}\text{C}$. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. **CAUTION:** Do this addition under the hood, Cl_2 could be evolved. Calibration working standards are prepped with each batch of samples and undergo a digestion of at least 30 minutes. Soil standards are brought up to 100mL final volume with DI H_2O prior to analysis.

8.2.2.2 Aqueous Calibration standards, add approximately 90 mL DI H_2O to each BOD bottle. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3 , mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. Do not volumize to 100 mL with DI H_2O prior to analysis. **CAUTION:** do this addition under the hood, Cl_2 could be evolved

8.2.3 A Calibration Blank, ICB/CCB, is prepared as the S0 calibration standard.

Sample concentrations are not reported below the lowest non-zero calibration standard of 0.2 $\mu\text{g/L}$ (aqueous samples, or 0.033 mg/Kg for soils) without method modification. This effectively becomes the method Reporting Limit

(RL) also referred to as Project Quantitation Limits (PQL). DoD QSM refers to this limit as the Limit of Quantitation (LOQ).

8.2.4 ICV/CCV Intermediate standards are prepared at 5.0 μ g/L from an **Independent Source** Hg Primary stock standard. The intermediate Hg CCV standard is prepared by pipetting 50 μ L of 1000mg/L Hg into a 100mL volumetric flask. The standard is brought to volume with 3%HCl for a final concentration of 500ug/L.

8.2.5 The working ICV/CCV is prepared by pipetting 1mL of the above intermediate standard into a BOD bottle and digesting as in **section 8.2.2**. An ICV/CCV is prepped with each batch of samples. Final concentration is 5 μ g/L and the standard is labeled as a mercury working calibration standard:

Label standard as IWyymmdd#, where:

I W = Inorganic Working

yy = year of preparation

mm = month of preparation

dd = day of preparation and

= Sequential letter of intermediate standards prepped on this day.

8.2.6 LCS/Matrix spike standard is also an intermediate concentration standard that is prepared by combining 45.5 uL of the **Independent Source** Hg Primary stock standard into a 100 mL volumetric flask and volumized to 100 mL with 3% HCl. The standard is labeled as for Hg working standards, as in **section 8.3**.

8.2.7 1mL of the above LCS/Matrix spike is added into 100 mL of DI H₂O and digested as an aqueous sample for the LCS-Water (LSCW) as in **section 8.1.1** . The true value of the LCS = 4.55 μ g/L at the instrument level.

8.2.8 1mL of the LCS/spike working spike is added to 0.6 g of acid-washed Teflon chips used to simulate a soil matrix for the LCS-Soil (LCSS). The true value of the LCS = 4.55 μ g/L at the instrument level.

8.2.9 1mL of the LCS/spike working spike is added to a chosen sample, and digested as per the sample matrix. This is the Matrix Spike. At the time of analysis, the spike = 4.55 μ g/L at the instrument level, times any sample dilution factor.

8.3 Instrumental Analysis

- 8.3.1 Adjust the argon pressure to 44psi for FIMS analysis. Turn on the FIMS system with the auto-sampler. Turn on the computer, printer. Allow the lamp to stabilize for 35-45 minutes.
- 8.3.2 Prepare the reductant solution, stannous chloride in **section 7.4**, and the carrier solution, 3% HCl in **section 7.11**.
- 8.3.3 Bring up the appropriate element file (Hg comm).
- 8.3.4 Set up the sample info file to coincide with the locations and sample identifications that will be analyzed in the run.
- 8.3.5 Fill out the Automated Control Window:
 - 8.3.5.1 Type in a data file name.
 - 8.3.5.2 Type in the name of the sample info file.
- 8.3.6 Load the auto-sampler tray.
- 8.3.7 Place the carrier tubing inlet into the carrier solution, and the reductant tubing inlet in the reductant solution. The reductant solution tubing has a red tab on it, and the carrier solution a yellow tab. Remove the cap from the gas chamber and start the pump to ensure it is working properly. Turn off the pump and replace the cap.
- 8.3.8 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the instrument run logbook.
- 8.3.9 On the AS-90 control window either choose "Run All" to run both standards and samples or "Calibrate" to run only the standards. If you choose only to calibrate at this time, you will need to click on "Reset Sampler" and "Run Samples" when calibration is complete. Analyze standards and samples. Samples that exceed the upper calibration range must be diluted and reanalyzed.
- 8.3.10 When analysis is complete place both carrier tubing inlet and the reductant tubing inlet with the auto-sampler probe in a beaker of DI water. Allow the water to pump through the system. Continue to flush all the water through until no more bubbles appear in the waste tubing. Turn off the pump.

8.3.11 Turn off the FIMS unit.

8.3.12 No rinse between samples is necessary per manufacturer's instructions.

8.3.13 The following Analytical Sequence should be used.

1. Calibration Blank(S0)
2. Standard #1 (S1)
3. Standard #2 (S2)
4. Standard #3 (S3)
5. Standard #4 (S4)
6. Standard #5 (S5)
7. ICV
8. ICB
9. Method blank
10. LCS
11. Samples (≤ 6)
12. CCV
13. CCB
14. MS
15. Sample Duplicate
16. Samples (< 5)
17. CCV
18. CCB

8.3.14 All information including the analytical sequence is documented in the FIMS 100 Run Logbook, **Attachment 2**.

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of ug/L for aqueous samples, and ug/Kg dry weight for soils/solids. Results are reported to two significant figures.

9.1.1 For aqueous results, report the data generated directly from the instrument with allowance for sample dilution. Upload the data from the FIMS directly into the Omega LIMS system for reporting.

9.1.2 For soil/solid samples, upload the data as for aqueous samples. The LIMS will calculate the final results in soil units, with allowance for dilution, sample weight and percent moisture. Make sure the Pmoist data is available.

9.2 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery } (\%R) = 100 \times (\text{SSR} - \text{SR}) / (\text{SA})$$

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.3 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$\text{RPD} = \frac{(\text{D1} - \text{D2})}{(\text{D1} + \text{D2})/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10 Quality Assurance/Quality Control

- 10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.
- 10.2 Linear correlation for the standard curve must be ≥ 0.995 . The Calibration curve must not be forced to go through the origin zero point.
- 10.3 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method Blanks must not contain Mercury at a concentration \geq the MRL. If mercury is present in the Method Blank and the lowest sample concentration in the batch exceeds 10 times the blank concentration, no corrective action is needed. Otherwise, corrective action for method blank contamination involves determining the source of the contamination and re-prepping the entire batch.

DoD QSM - Method blank concentrations must be less than 1/2 RL.

- 10.4 Calibration verification – A second source ICV prepped with the associated samples is analyzed immediately after the curve and must be within 90-110% of its true value. The ICV concentration is at the mid-level of the calibration curve.

- 10.5 CCVs are analyzed at least every 10 samples with 80-120% recovery requirements.
- 10.6 An ICB/CCB is run immediately after the ICV/CCV set; the mercury value in the ICB/CCB is not to exceed the MRL.

DoD QSM– Mercury is not to exceed the LOD in the ICB/CCB.

- 10.7 A matrix spike and a matrix duplicate are prepped and analyzed with every batch not to exceed 20 samples. The RPD for duplicates is 20% and the aqueous spike recovery control limit is 75-125%. The soil spike recovery control limit is 80-120%.

DoD QSM – Spike recoveries must be within 80-120% for both water and soil

- 10.8 Laboratory Control Sample (LCS) -An LCS is prepped with a minimum of every 20 samples of the same matrix. Control limits are 80-120% of the true value for mercury. If the LCS is outside the acceptance limits, the corresponding samples are re-prepped and reanalyzed. Corrective action includes re-digestion/reanalysis for all samples and QC in the batch.
- 10.9 Sample concentrations that exceed the highest calibration are diluted and rerun so that their concentration falls within the calibration range.
- 10.10 The Inorganic Laboratory Supervisor/Manager authorizes any method deviations.
- 10.11 The intermediate standard is stable for 28 days. All standards made from a primary standard expire on or before the primary standard's expiration date.
- 10.12 Method detection limits (MDLs) are established when the instrument is set up or when there is significant instrument maintenance performed that would affect its sensitivity. The MDL is obtained by multiplying the standard deviation of seven or eight analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. An MDL verification check is performed immediately following MDL study and quarterly each year in lieu of the annual MDL study. An MDL verification check sample is spiked at approximately 2-3 times the current MDL.

DoD QSM – The MDL verification check sample's concentration sets the LOD.

- 10.13 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the maintenance logbook. Trends are to be monitored to show the instrument is within the optimal absorbance range. The normal/optimal absorbance range is from 0.17-0.24.

11. Data Validation and Reporting

- 11.1 Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the Supervisor/Lab Manager. The Supervisor/Lab Manager reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. After each review, the appropriate section of the Data Review Checklist is checked off, **Attachment 3**.
- 11.2 Reports are generated by the data reporting group. The data submitted for report preparation is dependent on project requirements.
- 11.3 Data is always reported to the RL. If clients require reporting limits different from the RL/PQL, the data will need to be reported using a special form set through the Omega LIMS system depending on project needs.
- 11.4 Electronic files (EDD) are generated by the Data Management Department and are stored in the server until downloaded to tapes by the server backup system.

12. Corrective Action Procedures

- 12.1 Corrective Action to be implemented in the event QC results are outside of the acceptance range is covered in **section 10**.
- 12.2 Corrective Action reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a corrective action report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

- 13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Manual. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

- 13.2 Concentrated nitric, sulfuric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.
- 13.3 The cell-heating compartment maintains a temperature of 100°C throughout the analysis. Care should be taken to avoid burns from the cell-heating compartment.
- 13.4 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency: Test Methods for Evaluating Solid Waste, Update IIB, SW-846 Method 7470A and Update IV SW-846 Method 7471B.
2. Department of Defense, Quality Manual for Environmental Laboratories. Final Version 4.1, 4/22/09.

Attachments:

Attachment 1: Mercury Digestion Logbook

Attachment 2: FIMS Instrument Logbook

Attachment 3: Data Review Checklist Form

Attachment 1
Mercury Digestion Logbook

Attachment 2
FIMS Instrument Logbook

MITKEM LABORATORIES: FIMS 100 RUN LOGBOOK

Date: _____

Analyst: _____

Analysis:			Analysis:			Analysis:			Analysis:		
SEQ#	Bottle ID	LAB ID									
1			26			51			76		
2			27			52			77		
3			28			53			78		
4			29			54			79		
5			30			55			80		
6			31			56			81		
7			32			57			82		
8			33			58			83		
9			34			59			84		
10			35			60			85		
11			36			61			86		
12			37			62			87		
13			38			63			88		
14			39			64			89		
15			40			65			90		
16			41			66			91		
17			42			67			92		
18			43			68			93		
19			44			69			94		
20			45			70			95		
21			46			71			96		
22			47			72			97		
23			48			73			98		
24			49			74			99		
25			50			75			100		

Calibration/ICV Prep Date: _____

SnCl2 _____

HCl: _____

Reviewed By: _____

NaCl: _____

Hydroxylamine Sulfate: _____

Attachment 3
Data Review Checklist Form

MITKEM LABORATORIES

CLP/CLP-like Deliverable Check List for Inorganic Analysis

Project Number: _____
 Client: _____
 Input by/date: _____
 Forms generated on/date: _____

Analysis: _____
 Category: _____
 Reviewer: _____
 (1) Date Reviewed: _____
 (2) Date Reviewed: _____
 Corrections by: _____

Elements Required:

Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Ni	K	Se	Ag	Na	Tl	V	Zn	Sn	CN	Hg

<u>Items:</u>	<u>Pages</u>	<u>Check</u>	<u>OK/Unusual Observation</u>
Sample Log-In Sheet	_____	_____	_____
Prep Log Sheet (AQ/SL)	_____	_____	_____
% Solid Bench Sheet	_____	_____	_____
Tumbling Log (TCLP/SPLP)	_____	_____	_____

	<u>Check</u>	<u>Lab ID</u>	<u>OK/Unusual Observation/Deviation/Flags</u>
ICV / CCV	_____	_____	_____
Matrix Spike (N)	_____	_____	_____
Duplicate Samples (*)	_____	_____	_____
Serial Dilutions (E)	_____	_____	_____
Post Digestion Spike	_____	_____	_____
LCS	_____	_____	_____
ICP Interference	_____	_____	_____
CRA / CRI	_____	_____	_____

Prep/Analysis Notes:

	<u>Yes</u>	<u>No</u>
Client ID Check:	_____	_____
ID Truncation:	_____	_____
Special Request:	_____	_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

SOP No. 110.0038

Rev. 7

Signature

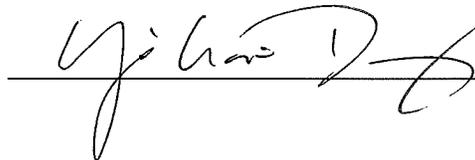
Date

QA Director:



2/19/09

Lab Director:



2/19/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

Rev. 7

1. Scope and Application

This Standard Operating Procedure (SOP) describes the procedure for measuring the percent solids/percent moisture of many types of solid material. An undried portion of sample is weighed and dried at 105 ° C. The sample is cooled, reweighed and the percent of water evaporated provides the result.

Note: It may be necessary for some specifically identified samples to undergo a pre-drying process. See amendment.

2. Personnel Qualifications and Responsibilities

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. Analysts and technicians are responsible for performing their work tasks following the procedures outlined in the SOP. Supervisors and managers are responsible for reviewing and approving new and revised SOPs. They are responsible for ensuring that SOPs are accurate and up to date, and are being implemented appropriately.

3. Summary of Procedure

Any standing water in the sample container is decanted. Samples are mixed to ensure sample homogeneity. Percent solid is calculated by the evaporation of water in a solid sample to a constant weight in a 103-105 ° C oven. .

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are stored prior to percent solids determination at a temperature between 2-6° C.
- 4.2 There is no “official” sample holding time. This analysis should be performed as soon as possible after sample receipt.

5. Interferences and Potential Problems

- 5.1 Temperature fluctuations will cause measurement errors.
- 5.2 Balance fluctuations can cause measurement errors. Also, if the balance is not properly calibrated, errors can occur.
- 5.3 Negligence in taring the balance prior to recording weights will also cause errors.
- 5.4 Results of less than 50% solids for ISM01.1 soils from EPA require immediate SMO notification and guidance.
- 5.5 If a SOM01.2 soil sample has greater than 65% moisture, the laboratory may use up to 50 g of soil sample in order to achieve the expected CRQLs. The amount of sample used and the % moisture should be noted in the SDG Narrative

6. Equipment and Apparatus

- 6.1 Aluminum weighing pans.
- 6.2 Top Loading balance, capable of reading to two decimal places, connected to a PC loaded with the Balance Talk software program.
- 6.3 PMOIST Binder to store photocopies of the Balance Talk worksheets
- 6.4 Scoopulas for portioning the sample.
- 6.5 Metal pans for moving samples in and out of the oven.
- 6.6 Class “S” or Class “2” weights for balance calibration.
- 6.7 Drying oven capable of maintaining 103-105 ° C (ISM01.1 allows 105 ± 5° C).

6.8 Class "1" weights for monthly calibration/verification .

6.9 Dessicator.

7. **Reagents**

Not applicable.

8. **Procedure**

Sample Prep: *(It maybe necessary to "pre-dry" some samples. See amendment, sec. 16.)*

8.1 Run the PMOIST backlong in LIMS by due date. Select the samples for PMOIST.

8.2 In LIMS, go to the Data Entry page. In the data entry page, click on **Export to Excel**. In the **Test Code** field, select PMOIST.

8.3 Remove the check marks from the **MBLK** and **LCS**, and select the **Excel** button. **SAVE** the file as a temporary excel file and select the **OPEN** button. When prompted for confirmation, select the **YES** button. The temporary excel file should be named *aaayymmddx*: where aaa are the initials of the person performing the PMOIST, yy is the last two digits of the year, mm is the month, dd is the day and x is A, B, C, etc., which is the sequence number. For example, the second temporary spreadsheet on January 14, 2009 would be called arh090114B.

8.4 In the temporary spreadsheet, delete any samples in which PMOIST will not be performed yet.

8.5 Add any sample DUPs now. Sample DUPs are performed every 20 samples. To add a sample DUP, highlight the row with the sample that a duplicate analysis is to be performed upon. Copy the highlighted row and select Insert Copied Cells option. Add the suffix DUP to the end of the ID for the sample that was just copied.. Be sure to save the changes made.

8.5.1 Duplicate samples for ILM05.4 projects must be done on the same sample that is chosen for Matrix Spike. The Duplicate analysis result is actually reported on Form 6 of the data package.

- 8.6 Label the aluminum weighing dishes with the Mitkem sample ID .
- 8.7 Minimize the Omega screen and the saved temporary spreadsheet. On the desktop, double click on the My Network Places icon. Double click on Public in Avogadro, Lims and then Pmoist. Scroll down and select file newestPmoist. If a macro warning box is displayed, choose the button **Enable Macros**.
- 8.8 Using the **Save As** option in the File Menu save the opened workbook using the following notation: YY =last two digits of the year; MM = numeric month example (June = 06, July = 07) DD = numeric value of the date of the month, X = letter corresponding to sequence number for the day. Do not forget to save this file to the Avogadro/ Public/Lims/**PMoist** directory.
- 8.9 Move to the **Header Information** page. Select the **Clear Data** button to remove any old data remaining from a previous batch. Complete the **Date and Time In/Out** and **Temperature** fields, and save to the new **PMoist** workbook.
- 8.10 Copy Mitkem Sample IDs from the temporary Excel spreadsheet starting with cell A2 to D2 down to Ax and Dx, where x is the number of the last row of samples.
- 8.11 Paste Mitkem Sample IDs into the Sample field starting in cell (A2) of the Data Entry page of the new **PMoist** spreadsheet. The temporary spreadsheet should be closed at this point.
- 8.12 The balance calibration must be checked and recorded in the appropriate logbook. Use calibration class "S" check weights of 1.0, 10.0 and 100 grams. If the balance does not meet the calibration check criteria listed in the logbook, do not proceed, but inform the supervisor.
- 8.13 In the spreadsheet move the cursor to cell H2 (first cell under Tare Weight column). Begin to collect the weight of the weighing tin: make sure the balance is zeroed. Place a weighing tin on the balance. When the balance displays the ""*"" press the "print" button on the balance. The tare weight is then printed in the highlighted cell of the spreadsheet. The cursor automatically goes to the next cell (Tare + Sample).
- 8.14 Open the sample jar. If there is any free water standing on the surface of the sample, this is carefully decanted (poured) into a waste container. Using a clean scoopula, the sample in the jar is mixed to insure it is homogeneous (well mixed).
- 8.15 Collect the weight of the wet sample and weighing tin. Measure out approximately 5-10 grams of sample. The weights are recorded to the nearest 0.01 gram. When the balance displays the ""*"" press the print button on the balance.

The weight of the weighing tin and sample is then printed in the highlighted cell (Tare + Sample). The cursor automatically goes to the next Tare Weight cell

- 8.16 Proceed with steps 8.13 to 8.15 until all samples are done. The spreadsheet should be saved periodically.
- 8.17 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.
- 8.18 As each sample is weighed, its tin is placed on a metal tray. As the tray of samples is complete, it is carefully placed on a shelf in the 103-105 ° C drying oven. Samples are dried overnight, optimally for at least 12 hours. ILM5.4/ISM01.1 soils have a 12 hour minimum drying time, and a 24 hour maximum.
- 8.19 If for some reason the samples cannot be dried overnight, a shorter drying time may be used, provided the sample is dried to a demonstrated constant weight. This procedure can only be used with the Supervisor's approval and must be carefully documented. The weighed wet sample is placed in the drying oven for at least four hours. Following four hours, the sample may be removed from the oven, cooled in the desiccator, weighed, and returned to the oven. After an additional 30 minutes of drying in the oven, the sample may be removed, cooled, reweighed, and replaced in the drying oven for an additional 30 minutes of drying. If two consecutive weights are equal (within 4%) then the sample has been dried to a constant weight, and the percent solids may be calculated.

This procedure must be documented in the PMOIST binder. A note is made for the sample that undergoes this procedure. The reverse side of the page is used to record the initial wet sample weight and time sample is first placed in the oven. When the sample is removed for weighing, the time and weight are also recorded on this page. There must be at least three weights and times recorded (initial and three consecutive constant weights).

ILM5.4/ISM01.1 samples which cannot be dried for the full 12 hours must also have demonstration of a constant final weight. Data must be recorded for a minimum of two repetitive 'weigh/dry/desiccate/weigh' cycles with a minimum of 1-hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

- 8.20 DAY TWO: Open the saved **PMoist** spreadsheet from the previous day. Enter date and time the samples were taken out of the oven and the oven temperature. Place the cursor so that it is in cell J2 (Final Wt). Ensure the balance has been calibrated and is zeroed as described above.

- 8.21 The tray of samples is carefully removed from the oven and the samples are placed in a dessicator to cool.
- 8.22 Put the cooled, dried sample and pan on balance. When the balance stabilizes press the **Print** button on the balance.
- 8.23 Select the **Calculate Pmoist** button. Click again on the Data Entry sheet of the worksheet if the calculations appear acceptable. On the header information page select the **Copy Data** button. Click on the **Print Report** button. Save and close the Excel program.
- 8.24 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.
- 8.25 From the main page of Omega click on the **Data Entry** button.
- 8.26 Select the **Add** button. From the instrument ID dropdown box select Dry Weight Oven (DWO). Complete the Run Start Date field. From the Analyst field dropdown box select the analyst name. Select the **Data Import** button.
- 8.27 From the **Specification** dropdown box select **Omega Excel File**. Select the **Run Import** button. From the **Open Dialog** box select the **PMoist** file and then select the **Open** button. Leave the value of 1 in the Text box and then select the **OK** button in the Spreadsheet Range box.
- 8.28 After Omega finishes importing the data, select the Data button. Of the three fields only the raw field should have value. Select the **Calc Seq** button and then select **Yes** when asked to continue.

9. **Data Reduction and Calculations**

The software performs all calculations. The results are reviewed by an independent analyst and then verified in the LIMS Omega System.

10. **Quality Assurance/Quality Control**

- 10.1. A duplicate sample must be performed at least every 20 samples.
- 10.2. The balance calibration must be within specifications.
- 10.3. The balance must be calibrated monthly with the Class "1" weights located in the QA/QC Office.

10.4. The drying oven temperature must be stable in the range of 103-105° C.

10.5. Times samples are put into and taken out of the oven are recorded.

11. Data Validation and Reporting

Results are checked first by the analyst and then by the supervisor. The print-out of the completed Excel spreadsheet is delivered to the Supervisor or designee for review.

Following Supervisor review and approval, all data are QA Validated in the Omega LIMS. The Excel spreadsheet is dated and sequentially numbered and scanned into the PMOIST optical file database for permanent storage. The original hardcopy record is saved in the PMOIST binder.

12. Corrective Action Procedures

12.1 If the balance calibration exceeds acceptance criteria, recalibrate it according to manufacturer's instructions.

12.2 If the drying oven shuts down during the night, the analysis will need to be restarted, but samples will not need to be re-weighed.

12.3 If drying oven temperature exceeds 110° C, samples will need to be re-prepped and reanalyzed.

12.4 If a sample spills during the final weighing, it will need to be re-prepped.

12.5 If a sample and duplicate RPD vary more than 35%, another independent analysis of the sample must be performed.

12.6 If sample drying time is not within 12 to 24 hours for ILM/ISM soils, the time must be noted for inclusion in the project narrative.

13. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of samples used in the method. Lab coat, gloves and safety glasses must be worn at all times in the lab.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

APHA, AWWA, and WEF, "Standard Methods for the Examination of Water and Wastewater," 18th edition.

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Organic Analyses (Multi-Media, Multi-Concentration) SOM01.2

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Analyses (Multi-Media, Multi-Concentration) ILM05.4

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM01.1

Balance Talk user manual

16. Amendment to SOP

16.1 Due to some sample matrixes and/or special client requests, it may be necessary to "pre-dry" samples prior to the sampling procedure. These samples will be specifically identified by the Laboratory Supervisor or Project Manager.

16.2 Visually determine or weigh out approximately 15 – 20 grams of each sample in an appropriate container. (An aluminum pan should be sufficient)

16.2.1 Place each sample in a drying oven set at 60°C overnight.

16.2.2 Follow the procedure as described in **Section 8**.

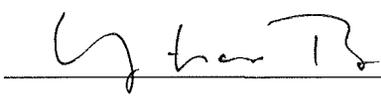
**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B)

**SOP No. 50.0052
Rev 3**

	Signature	Date
QA Director:	<u></u>	<u>2/2/10</u>
Lab Director:	<u></u>	<u>2/4/10</u>
Effective Date:	<u>02/11/10</u>	

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

**Organic Preparation of Soil Samples by Sonication
(Method 3550B)
Rev 3**

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples using ultrasonic extraction for analysis by EPA SW846 methods SW8081, SW8082, SW8015 and SW8270. Discussions include sample extraction, sample cleanup references, and the preparation of standard spiking solutions for the analysis of Total Petroleum Hydrocarbons (TPH), Diesel Range Organics (DRO), pesticide/PCB or semivolatile organic compounds in soil samples. Highly contaminated soils for MA DEP EPH analyses may also use this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. The sample is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using TurboVap or Kuderna-Danish (KD) apparatus followed by Nitrogen blowdown. In addition, this method can be used for Medium/High Concentration Semivolatile samples. A 1-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted once using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection.
- Sample extracts must be analyzed within 40 days of sample extraction.

4.2. Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3. Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1. Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2. Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3. Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1. Ultrasonic Disrupter – pulsing horn type with 375 watts maximum and ¾” standard solid disrupter horn and ½” standard tapered microtip probe (medium soils only). Fisher Scientific Sonic Dismembrator Models 500 and 550.

6.2. Sonobox – ultrasonic disrupter box designed to reduce exposure to ultrasonic sound.

6.3. Beakers – 400mL

6.4. 500mL Erlenmeyer flask

6.5. Dessicator

6.6. Balance capable of weighing +/- 0.1 gram.

6.7. Glass syringes for delivering spike and surrogate solutions – 0.25mL, 0.5mL, 1.0mL.

6.8. Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.9. Aluminum Foil (Industrial Grade)

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvents and standards are extensively used in the lab:

7.1. Methylene chloride: pesticide quality or equivalent to be used for glassware rinsing and sample extraction.

7.2. Methanol: pesticide quality or equivalent, to be used for rinsing glassware and preparing standards.

7.3. Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4. 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5. Hexane: pesticide quality or equivalent , to be used for solvent exchange of samples.

7.6. H₂O, deionized.

7.7. H₂SO₄, concentrated, for sample pH adjustment.

7.8. Surrogate Standards *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5- α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9. Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

7.10. Anhydrous sodium sulfate: granular for drying the samples and sample extracts. Baked at 400°C for four hours.

8. Procedure

8.1. Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1. All **primary standards** received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB

P= Primary standard

yymmdd = date standard is received, and

X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2. Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and

diluting to 250mL using methanol.

The working standard contains the following compounds:

<u>Compound</u>	<u>Concentration(µg/mL)</u>
1,2-Dichlorobenzene-d ₄	50
2-Fluorobiphenyl	50
Nitrobenzene-d ₅	50
p-terphenyl-d ₁₄	50
2-Chlorophenol-d ₄	75
2-Fluorophenol	75
Phenol-d ₆	75
2,4,6-Tribromophenol	75

Semivolatiles SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.8** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

The working surrogate spike standard is labeled SWyymmddX where:
 SW = Semivolatiles working standard
 yymmdd = date working standard is prepared,
 X = the order that the working standard is prepared on that date, in increasing alphabetical order.

- 8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8**, and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

<u>Compound</u>	<u>Concentration(µg/mL)</u>
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

- 8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by taking 1mL of the 5- α Androstane and 1mL of the o-Terphenyl primary standards in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains the following compound:

<u>Compound</u>	<u>Concentration(µg/mL)</u>
5- α Androstane	50
o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3. Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 5.0 mL of 8270 MegaMix, 2.5mL of 3,3' DCB, and 5.0 mL of 8270 add-on intermediate standard from **section 7.9**, and diluting to 50mL using methanol. The resulting concentration will be 50ug/mL for all SW8270 compounds. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2. The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See **Attachment 1** for a list of the individual compounds and their concentrations.

8.1.3.3. The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

<u>Compounds</u>	<u>Concentration(µg/mL)</u>
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.5. The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10mL Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX
where:

SI = Semivolatile intermediate standard

yymmdd = date intermediate standard is prepared,

X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1mL of the

intermediate standard above, and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following:

<u>Compounds</u>	<u>Concentration(µg/mL)</u>
Diesel fuel	5000

The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section 7.9 and 2.5mL of the Aromatic Hydrocarbon standard in Section 7.9 and diluting to 250mL in 1:1 methylene chloride: acetone mixture. The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1.**

A smaller or larger amount may be used to prepare the Lab Control Sample and Matrix Spike Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at -10 to -20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at -10 to -20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.1.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2. Sample Extraction Procedure:

- 8.2.1. Low Level Method: Unless Mitkem has documented history that the samples contain high concentration of target analytes (>10,000µg/Kg for the individual analyte) all of the semivolatile samples are prepared using the low-level approach.

Before starting:

- The disrupter has a minimum of 375 watts power and must be inspected periodically to ensure that the ¾ inch tip has not experienced excessive wear.
- The disrupter is tuned (or verified as with the Fisher 500) each day of use. Follow manufacturer's guidance for tuning procedures (see instructions

posted by Sonicators). The tuning is documented in the Sonicator Horn Tuning Logbook, **Figure 4**. After tuning, note in extraction logbook as well.

- Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.1.1. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400mL glass beaker. A 30 gram \pm 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopula. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

NOTE: A smaller sample size may be used as long as all surrogates and final volumes are modified appropriately. Associated QC samples should be adjusted similarly. Where appropriate or allowed, using a smaller sample volume can help to reduce solvent use and thereby minimize solvent waste.

8.2.1.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing; however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.1.3. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes. According to the test method being used, add the following spikes to the samples in beakers:

- Use a syringe to add 1.0mL of the surrogate standard to the Blank, LCS, samples and any MS/MSD sample.
- Use a syringe to add 1.0mL of the LCS/MS standard to the LCS, and any MS/MSD sample.

8.2.1.4 Add 100mL of the 1:1 v/v methylene chloride/acetone mixture to the Blank, LCS, samples, and any MS/MSD for Semivolatile or Pesticide/PCB extraction, OR add 100mL of methylene chloride to the Blank, LCS, samples, and any MS/MSD for TPH/DRO extraction.

8.2.1.5 Place the bottom surface of the $\frac{3}{4}$ inch tip of the disrupter about $\frac{1}{2}$ inch below the surface of the solvent, but above the solid layer.

8.2.1.6 Extract ultrasonically for 3 minutes, with the output control set at full power and the mode switch on Pulse, using a 50% duty cycle.

8.2.1.7 Remove the beaker from the disrupter. Decant the solvent layer into a

500mL Erlenmeyer flask, leaving the solid in the beaker.

- 8.2.1.8 Extract the sample two more times with 100mL of solvent and a 3 minute sonication time. Decant the solvent aliquots into the 500mL Erlenmeyer flask. Cover the flask with aluminum foil.
- 8.3. Semivolatile Medium Level Method: (Also refer to **SOP No. 50.0100** , Method SW846 3570 Microscale Solvent Extraction for another soil extraction option using small sample volumes.)
 - 8.3.1. The sample is mixed to ensure sample homogeneity. As representative as possible, weigh about a 1 gram portion of the sample into a pre-weighed 15mL vial. The weight measurement is recorded to the nearest 0.1 gram.
 - 8.3.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. Mix the sample and anhydrous sodium sulfate to achieve a free flowing mixture.
 - 8.3.3. Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.
 - 8.3.4. Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples.
 - 8.3.5. Add 9mL of methylene chloride to the Blank and samples. Add 8mL of methylene chloride to the LCS and any MS/MSD.
 - 8.3.6. Sonicate the sample with the 1/8 inch tapered Microtip for 2 minutes at output control setting at 5 in the continuous mode.
 - 8.3.7. Cap the vial.
 - 8.3.8. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).
- 8.4. The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.
- 8.5 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:
 - 8.5.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to SOP No. 50.0030 Method 3660B Sulfur Cleanup, for details on activating copper and using it in a sample extract.

- 8.5.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to SOP No. 50.0031 Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.
- 8.5.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP No. 50.0032 Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.
- 8.5.4. Other cleanup methods may be found in SOP numbers 50.0033(Florisol) and 50.0034 (Silica Gel).
- 8.6 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.
- 8.7 Sample and Extract Disposal:
- All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.
- 8.7.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.
- 8.7.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1. Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2. Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- An aliquot of the surrogate standard prepared in **section 8.1.2** is added to the Method Blank.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2. Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2. Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the

corresponding method blank.

- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1. Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4. Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan; Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

Attachments:

Attachment 1: Pesticide LCS/MS List

Attachment 2: Sonicator Tuning instructions

Figure 1: Semivolatile Extraction Log

Figure 2: Pesticides/PCB Extraction Log

Figure 3: Re-extraction Request

Figure 4: Sonicator Tuning Log

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4'-DDD	0.4
4,4'-DDE	0.4
4,4'-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Attachment 2
Sonicator Tuning instructions

The User I/O may be used to remotely control the system. If this is the case, you must design in whatever safety precautions are appropriate to your User I/O circuit design to prevent unexpected start-up, which can cause personal injury and can cause equipment damage.

3.8 Ultrasonic Test

The Test button on the front panel of the Model 500 is used to verify that the unit is functioning (providing ultrasonic energy to the converter and horn). You can also run another test on the system for your particular experiment as described later. Before testing the Model 500, always make sure that the horn is not touching anything. The system will perform several self-tests when it is first turned on.

Step	Do this...	To obtain this result
1	Set up the Model 500 following the instructions in this manual. If no horn is currently installed, mount a 1/2" disruptor horn to the converter.	Prepare the Model 500 to operate, if it was not previously assembled.
2	After you have connected the converter/horn to the converter cable, verified all other connections are as desired: Turn the unit on, and observe the self-test displays.	Verify that the system passes all its self-tests, observing that there are no error messages on the front panel display. The Model 500 advances to the ready mode and shows the normal "Ready" display.
3	Adjust the amplitude control to approximately 50% (observe the value on the front panel display).	Ensures that ultrasonic energy will be at some mid-range value, and will not cause damage if you were using a microtip (must be less than 70%).
4	Verify that the horn is not touching anything. Press the test button on the front panel. Observe the front panel display.	Verifies the ultrasonic output of the system. You may hear a soft, high-pitched sound. The bargraph display will show some output value. The test will run for 2 seconds, then stop.
5	If the system showed readings on the display during the test, you may either proceed with your experiments or turn the unit off.	Verification that the Model 500 Dismembrator is operating and is ready to be set up for your experiment.

Sonic Dismembrator

Fisher Scientific

Model 500

SOP No. 50,0052 Rev 3
 Date Initiated: 04/20/06
 Date Revised: 02/02/10
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F. TUNING INSTRUCTIONS - (for standard probes, Q-horns and cuphorns)

To assure optimum operation tune the generator in accordance to the following procedure each time a new probe is changed.

The probe or microtip should not be immersed in the liquid or come in contact with the work surface when tuning.

When operating with liquids at extreme temperatures, immerse the probe in the liquid for a few minutes, remove from the liquid then tune.

1. Turn OUTPUT CONTROL knob counter-clockwise to zero.
2. Press POWER SWITCH to ON (up) position. The switch will illuminate.
3. When the prompt [for tuning procedure refer to manual] appears, press TUNE key. Screen will read:
[TUNING - - - PROBE ACTIVE].
4. Turn the Output Control Knob towards setting 3.
 - a) Note the position of the Bar Graph on the ICD Display Screen. Do NOT exceed 70%.
 - b) Rotate the Tuning Knob clockwise or counterclockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.
5. Turn Output Control Knob towards setting 6.
 - a) Again, note the position of the Bar Graph and do not exceed 70%.
 - b) Rotate the tuning knob till you obtain a meter reading of 20% or below.
6. Repeat step 5 on power setting 10. Minimize the reading one last time to 20% or less.
7. Press the TUNE key to display prompt for programmed or continuous operation.

YOUR SONIC DISMEMBRATOR IS NOW TUNED

SPECIAL NOTE FOR TUNING MICROTIP™ PROBES AND OTHER HORNS

MICROTIPS

The above procedure must be followed with the exception that the OUTPUT CONTROL should NEVER exceed the MICROTIP LIMIT (5). Tuning at the MICROTIP LIMIT should be done as quickly as possible. Prolonged operation in air at the limit can cause MICROTIP failure.

CUPHORNS

Drain filled cups down to outlet fitting level, tune, and refill.

Sonic Dismembrator -9- Fisher Scientific
SOP NO. 50.0052 Rev 3 Model 550

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Figure 1
Semivolatile Extraction Log

Figure 2
Pest/PCB Extraction Log

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:

DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Figure 4:
Sonicator Tuning Log

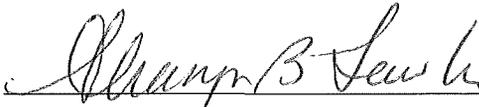
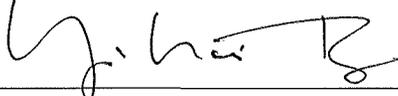
**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Soxhlet (Method 3540C)

**SOP No. 50.0053
Rev. 3**

	Signature	Date
QA Director:	 _____	<u>2/2/10</u>
Lab Director:	 _____	<u>2/4/10</u>
Effective Date:	<u>02/11/10</u>	

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

**Organic Preparation of Soil Samples by Soxhlet (Method 3540C)
Rev 3**

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples for analysis by EPA SW846 methods SW8081, SW8015, SW8082 and SW8270. Discussions include sample extraction, sample cleanup references, sample concentration technique and the preparation of standard spiking solutions for the analysis of semivolatile, Total Petroleum Hydrocarbons (TPH), Diesel Range Organics (DRO), or pesticide/PCB organic compounds in soil samples. Soil samples to be analyzed for MA DEP EPH may also be extracted using this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted overnight using soxhlet extraction. The extract is dried through powdered and granular anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Caliper TurboVap or Kuderna-Danish (KD) apparatus followed by nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection

- Sample extracts must be analyzed within 40 days of sample extraction
- 4.2 Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.
 - 4.3 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

- 5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.
- 5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.
- 5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

- 6.1 Beakers – 400ml.
- 6.2 Boiling flask – 500ml.
- 6.3 Soxhlet extractor.
- 6.4 Condensers, 55/50.
- 6.5 Heating mantle, 500ml.
- 6.6 Balance capable of weighing +/- 0.1 gram,.
- 6.7 Glass syringes for delivering spike and surrogate solutions – 0.25ml, 0.5ml, 1.0ml.
- 6.8 Volumetric flasks for making up surrogate and matrix spike solutions – 50ml, 250ml.
- 6.9 Aluminum Foil (Industrial Grade)

6.10 Hotplates (option instead of mantles)

6.11 Glass wool or filters

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvents and standards are extensively used in the lab:

7.1 Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction.

7.2 Methanol: pesticide quality or equivalent, to be used for rinsing glassware and preparing standards.

7.3 Acetone: pesticide quality or equivalent, to be used for rinsing glassware.

7.4 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5 H₂O, deionized.

7.6 H₂SO₄, concentrated, for sample pH adjustment.

7.7 Surrogate Standards: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086) at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL(Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.8 Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and*

traceable to reference materials.

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3, 3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1, 1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

7.9. Anhydrous sodium sulfate: granular for drying the samples and sample extracts. Baked at 400°C for four hours.

8. Procedure

8.1 Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes.

The standards are labeled ZPyymmddX,

where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB

P= Primary standard

yymmdd = date standard is received, and

X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is prepared by combining 2.5ml of the Base/Neutral primary standard in **Section 7.7** and 2.5ml of the Acid primary standard in **Section 7.7** and diluting to 250ml using methanol.

The working standard contains the following compounds:

<u>Compound</u>	<u>Concentration($\mu\text{g/ml}$)</u>
1,2-Dichlorobenzene-d ₄	50
2-Fluorobiphenyl	50
Nitrobenzene-d ₅	50
p-terphenyl-d ₁₄	50
2-Chlorophenol-d ₄	75
2-Fluorophenol	75
Phenol-d ₆	75
2, 4, 6-Tribromophenol	75

Semivolatile SIM_surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.7** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo (e) pyrene d-12.

The working surrogate spike standard is labeled ZWyymmddX where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB

W=Working Standard

yymmdd = date working standard is prepared,

X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.7**, and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

<u>Compound</u>	<u>Concentration($\mu\text{g/mL}$)</u>
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by

taking 1mL of the 5- α Androstane and 1mL of o-Terphenyl primary standards in Section 7.7 and diluting to 200 ml using methanol. The working standard contains the following compound:

<u>Compound</u>	<u>Concentration(μg/ml)</u>
5- α Androstane	50
o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 2.5ml of Base/Neutral Composite Set primary standard in **Section 7.8**, 2.5ml of Acid Composite Mix primary standard in **Section 7.8** and 2.5ml of Composite Mix #3 primary standard in **Section 7.8** and diluting to 100ml using methanol. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2 The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 μ g/mL. See Attachment 1 for a list of the individual compounds and their concentrations.

8.1.3.3 The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.8** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

<u>Compounds</u>	<u>Concentration(μg/mL)</u>
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.4 The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10ml Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX

where:

SI = Semivolatile intermediate standard

yymmdd = date intermediate standard is prepared,

X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1ml of the intermediate standard above, and diluting to 100ml using Methanol. The working LCS/MS Standard contains the following:

<u>Compounds</u>	<u>Concentration (µg/ml)</u>
Diesel fuel	5000

8.1.3.5 The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section 7.8 and 2.5mL of the Aromatic Hydrocarbon standard in Section 7.8 and diluting to 250mL in 1:1 methylene chloride: acetone mixture. The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1**.

A smaller or larger amount may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at -10 to -20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at -10 to -20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

Before starting:

- The soxhlet extractor must be inspected periodically to ensure that the glassware does not have any cracks.
 - Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.
- 8.2.1 Add about 3 Teflon boiling chips to the boiling flask. Place the soxhlet extractor over the boiling flask. Be careful not to knock the extractor over. Use glass wool or filter paper to plug the hole in the soxhlet extractor.
- 8.2.2 The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400ml glass beaker. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction. A 30 gram \pm 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopula. The weight measurement is recorded to the nearest 0.1 gram. Smaller portions of samples may be used. Adjust the final volume accordingly to maintain the same ratios for reporting purposes.
- 8.2.3 Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing; however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.
- 8.2.4 Pour the sample and anhydrous sodium sulfate mixture into the soxhlet extractor. Be sure not to get any soil into the boiling flask.
- 8.2.5 Sonicate the surrogate standard and the lab control sample standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes.
- Use a syringe to add 1.0 mL of the surrogate standard to the Blank, LCS, all sample and any MS/MSD sample that are in the soxhlets.
 - Use a syringe to add 1.0 mL of the LCS/MS standard to the LCS and any MS/MSD samples.
- 8.2.6 Cover the sample with glass wool to ensure that none of the soil splashes. Add 400ml of the 1:1 v/v methylene chloride/acetone mixture to the soil.
- 8.2.7 The boiling flask is placed in the heating mantle or on hotplate. The soxhlet is chained so it will not tip over. The condenser is placed on top of the extractor. Start the water running through the condensers. Plug the heating mantles in or turn on the hotplates. Measure the cycle/hour rate and record this in the extraction logbook. Allow the sample to extract for a minimum of 18 hours.
- 8.2.8 At the end of the extraction, unplug the heating mantles or turn off the hotplates.

Allow the samples to cool to room temperature. Take the soxhlet out, drain the extraction solvent into the boiling flask and dump the soil mixture into solid waste container. Cover the boiling flask with aluminum foil.

- 8.2.9 Record all extraction information, including the Date and Time the soxhlet extraction began and ended, in the appropriate Extraction Logbook (**Figures 1 and 2**).
- 8.3 The sample extract is now ready for filtration and concentration. These procedures are found in **SOP No. 50.0054**, Extract Filtration and Concentration.
- 8.4. After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:
- 8.4.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to **SOP No. 50.0030** Method 3660B Sulfur Cleanup, for details on activating copper and using it in a sample extract.
- 8.4.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to **SOP No. 50.0031** Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.
- 8.4.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to **SOP No. 50.0032** Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.
- 8.4.4. Other cleanup methods may be found in SOP numbers **50.0033**(Florisol) and **50.0034** (Silica Gel).
- 8.5 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.
- 8.6 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

- 8.6.1 Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.6.2 Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2 Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1 Frequency of LCS:

A LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2 Procedure for LCS:

- The LCS is prepared in identical fashion as the associated samples; and in addition:
- 1.0mL of the surrogate solution prepared in **Section 8.1.2** and 1.0 mL of the lab control sample prepared in **Section 8.1.3** is added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.

10.3 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2 Procedures for Duplicate Matrix Spikes:

- The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition
- 1.0 ml of the surrogate solution prepared in **Section 8.1.2** and 1.0 ml of lab

control sample prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.

- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

- 11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A re-extraction request will be filled out (**Figure 3**).

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**).

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will instruct the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**).

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3540C, Ultrasonic Extraction, Revision 3, December 1996.

Attachments:

Attachment 1: Pesticide LCS/MS List

Figure 1: Semivolatile Extraction Log

Figure 2: Pest/PCB Extraction Log

Figure 3: Re-extraction Logbook

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4'-DDD	0.4
4,4'-DDE	0.4
4,4'-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1
Semivolatle Extraction Log

Figure 2
Pest/PCB Extraction Log

Figure 3
Re-extraction Logbook

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:

DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-06/08

Reviewed By: _____

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3570)

SOP No. 50.0100

Rev 2

	Signature	Date
QA Director:	 _____	<u>2/2/10</u>
Lab Director:	 _____	<u>2/4/10</u>
Effective Date:	<u>02/11/10</u>	

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

**Organic Preparation of Soil Samples by Microscale Solvent Extraction
(Method 3570)**

Rev 2

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil, sediment, tissues, biota and any sample considered solid in nature. Discussions include sample extraction, sample cleanup references, sample concentration technique references and the preparation of standard spiking solutions for the analysis of semivolatiles or pesticide/PCB organic compounds in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 2 to 20-gram sample is weighed directly into a 40 or 60-mL VOA vial. The sample is solvent extracted first with acetone. Then it is extracted three times with methylene chloride by either a manual shake approach or via rotation or spinning of the sample. For analysis of Pesticides, PCBs and Petroleum samples hexane rather than methylene chloride can be used. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Kuderna-Danish (KD) or TurboVap apparatus.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection
- Samples can be held for one year if they are frozen

- Sample extracts must be analyzed within 40 days of sample extraction
- 4.2 Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to SOP No. 110.0039 for more detail on sub sampling techniques.
- 4.3 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

- 5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.
- 5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest
- 5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

- 6.1 Rotary extraction apparatus
- 6.2 40 and 60 mL VOA vials and caps.
- 6.3 Balance capable of weighing +/- 0.1 gram.
- 6.4 Glass syringes for delivering spike and surrogate solutions.

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. The following chemicals including solvents and standards are extensively used in the lab:

- 7.1 Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction.
- 7.2 Methanol: pesticide quality or equivalent to be used for rinsing glassware and

preparing standards.

- 7.3 Acetone: pesticide quality or equivalent, to be used for rinsing glassware.
- 7.4 Hexane: pesticide quality or equivalent, to be used for solvent exchange of samples
- 7.5 H₂O, Deionized.
- 7.6 Copper, reagent grade purified. Fine or Granular.
- 7.7 H₂SO₄, concentrated, for sample pH adjustment.
- 7.8 Surrogate Standards:
- Restek BN Surrogate mix (Cat. No. 31086) at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
 - Deisel Range Organics (DRO)/TPH: 5- α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL(Made from neat source(Aldrich)).
 - Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.
- 7.9 Lab Control Sample and Matrix Spike:
- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
 - Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
 - PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
 - TPH/DRO: LCS uses Diesel/gasoline.

8. Procedure

- 8.1 Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

- 8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile, P for Pesticides or PCB

P= Primary standard

yymmdd = date standard is received, and

X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

- 8.1.2 Preparation of Surrogate standard:

- 8.1.2.1. The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

<u>Compound</u>	<u>Concentration(µg/mL)</u>
1,2-Dichlorobenzene-d ₄	50
2-Fluorobiphenyl	50
Nitrobenzene-d ₅	50
p-terphenyl-d ₁₄	50
2-Chlorophenol-d ₄	75
2-Fluorophenol	75
Phenol-d ₆	75
2,4,6-Tribromophenol	75

The working surrogate spike standard is labeled SWyymmddX where:

SW = Semivolatile working standard

yymmdd = date working standard is prepared,

X = the order that the working standard is prepared on that date, in increasing alphabetical order.

- 8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8**, and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

<u>Compound</u>	<u>Concentration(µg/mL)</u>
-----------------	-----------------------------

Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile working LCS/MS Standard is prepared by combining 2.5mL of Base/Neutral Composite Set primary standard in **Section 7.9**, 2.5mL of Acid Composite Mix primary standard in **Section 7.9** and 2.5mL of Composite Mix #3 primary standard in **Section 7.9** and diluting to 100mL using methanol. All compounds have a final concentration of 50 ug/mL.

8.1.3.2 The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See Attachment 1 for a list of the individual compounds and their concentrations.

8.1.3.3 The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

<u>Compounds</u>	<u>Concentration(µg/mL)</u>
Aroclor 1016	4.0
Aroclor 1260	4.0

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2**.

A smaller or larger amount may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at -10 to -20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at -10 to -20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross

contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.2**.

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

8.2.1. Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.2. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes.

8.2.3. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a 40 mL VOA vial. The sample is weighed into the tared VOA vial. The weight on the sample will vary with the testing procedure and matrix. Tissues usually are weighed in the 3-5 gram range. Soils and sediments are usually in the 5-7 gram range. The data quality objective of the work will dictate if there should be variations from these recommended target weights. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.4. Semivolatile Extraction Procedure:

8.2.4.1. Spike the samples and QC with the appropriate surrogates and spike solutions.

- Use a syringe to add 1.0mL of the Semivolatile surrogate standard to the Blank, LCS, samples and any MS/MSD sample that are in the vials.
- Use a syringe to add 1.0mL of the Semivolatile LCS/MS standard to the LCS and any MS/MSD samples.

8.2.4.2. Add 5-10 mL (7 mL) of acetone to Blank, LCS, samples, and any MS/MSDs. The volume should be enough to **completely cover the sample**. There are instances where samples will appear to absorb the solvent (humic materials for example). In these cases add enough solvent to cover the sample.

8.2.4.3. Cap the vials and shake vigorously for 30 seconds.

- 8.2.4.4. Allow the samples to settle after agitation.
- 8.2.4.5. Decant the solvent layer into a 60mL VOA vial, leaving the solid in the 40mL VOA vial.
- 8.2.4.6. If the samples are extremely wet sediments or tissues, repeat the acetone step from above. At this step, water has been removed from the sample by the acetone.
- 8.2.4.7. For sediments, sulfur is always suspected. Activated copper can be added to the acetone in the 60 mL vials to remove sulfur.
- 8.2.4.8. Extract the sample three more times with 5-10 mL (7 mL) of methylene chloride. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.
- 8.2.5. Procedure for Pesticides, PCBs and Petroleum Hydrocarbon:
- 8.2.5.1. Spike the samples and QC with the appropriate surrogates and spike solutions.
- Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.
 - Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples that is in the 40mL vial.
- 8.2.5.2. Continue as in **Sections 8.2.4.2 through 8.2.4.8.**
- 8.2.5.3. Extract the sample three more times with 5-10 mL (7 mL) of Hexane. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.
- 8.2.5.3.1. For the chlorinated Pesticides and PCBs which are readily extractable, the samples can be extracted by shaking the samples in hexane for three minutes rather than spinning the samples.
- 8.2.5.4. If the samples contain a petroleum odor, the sample size should be reduced to one to two grams.
- 8.2.6. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

- 8.3. Sample Filtration and Concentration: Refer to SOP 50.0054, Extract Filtration and Concentration.
- 8.4. Extract Cleanup: Refer to SOPs 50.0030 Method 3660B Sulfur Cleanup, 50.0031 Method 3665A Acid Cleanup, 50.0032 Method 3640A GPC Cleanup, 50.0033 Method 3620B Florisil Cleanup, and 50.0034 Method 3630C Silica Gel Cleanup for details on the different cleanup methods available.
- 8.5. The final extracts are transferred to the Semivolatile lab and documented in the Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.
- 8.6. Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

- 8.6.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.
- 8.6.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{(\text{Concentration of ampule})(\text{amount used})}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

- 10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2 Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1 Frequency of LCS:

A LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2 Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS Spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar

matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2 Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3570, Microscale Solvent Extraction, Revision 0, November 2002.

Attachments:

Attachment 1: Pesticide LCS/MS list

Figure 1: Semivolatile Extraction Log

Figure 2: Pesticides/PCB Extraction Log

Figure 3: Re-extraction Request

Attachment 1: Pesticide LCS/MS list

Analyte	Spike conc in ug/mL
4,4'-DDD	0.4
4,4'-DDE	0.4
4,4'-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1
Semivolatile Extraction Log

Figure 2
Pest/PCB Extraction Log

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:

DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-06/08

Reviewed By: _____

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

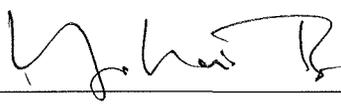
for

Organic Extract Filtration and Concentration Techniques

Rev 2

Signature

Date

QA Director:	<u></u>	<u>2/2/10</u>
Lab Director:	<u></u>	<u>2/4/10</u>
Effective Date:	<u>02/11/10</u>	

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques
Rev. 2

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the filtration and concentration of organic sample extracts that were prepared using EPA water and SW-846 solid hazardous waste methods, as well as OLC3.2, OLM4.3 and the current SOM version procedures.

2. Personnel Qualifications and Responsibilities

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. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

The sample extract is dried through granular anhydrous sodium sulfate and is made ready for cleanup and/or analysis following concentration using Kuderna-Danish (KD) apparatus or Caliper TurboVap apparatus and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

Not Applicable

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation and concentration; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

- 5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See Mitkem SOP Nos. 50.0030 through 50.0034 for cleanup methods.
- 5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation and concentration steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Equipment and apparatus used in this method include:

- 6.1 Teflon boiling chips.
- 6.2 Kuderna-Danish (KD) apparatus with a 10 or 15ml receiver tube.
- 6.3 80mm Glass funnel.
- 6.4 Glass wool.
- 6.5 Three ball Snyder column.
- 6.6 Water bath, capable of maintaining 60 °C to 90°C.
- 6.7 Boiling chips, carbon.
- 6.8 Nitrogen blow-down apparatus, N-EVAP Model No. 111.
- 6.9 Receiving vial with Teflon septa – 2ml.
- 6.10 15ml storage vials with screw caps.
- 6.11 Caliper TurboVap apparatus with 200mL collection tube
- 6.12 Aluminum Foil (Industrial Grade)
- 6.13 Glass Pipettes
- 6.14 20X150mm Disposable Culture Tubes

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the

accuracy of the test. The following chemicals including solvent, standards and gasses are extensively used in the lab:

- 7.1. Methylene chloride: pesticide quality or equivalent, to be used for glassware rinsing and sample extraction
- 7.2. Hexane: pesticide quality or equivalent, to be used for solvent exchange of samples.
- 7.3. Anhydrous sodium sulfate, granular for drying the sample extract.
- 7.4. Acetone: pesticide quality or equivalent, to be used for glassware rinsing.

8. Procedure

Note: Final extract volumes given below are those associated with 1000 ml or 30 g initial volume/wt samples. The final extract volumes for smaller initial volume/wt samples would generally be subjected to further concentration to maintain the same initial: final volume ratio. For example, a 15g PCB sample would be concentrated to a final 5 ml volume instead of 30g to 10 ml.

8.1 Sample Extract Filtration:

All sample extracts are dried through anhydrous sodium sulfate prior to further sample concentration.

- 8.1.1. A drying funnel is first cleaned with methylene chloride to remove any contaminants. The drying funnel is then prepared by plugging an 80mm diameter filtering funnel with glass wool and adding approximately 20 grams of anhydrous granular sodium sulfate.
- 8.1.2. Sample extracts are dried through the drying funnels and collected directly into the KD concentrators or Caliper TurboVap collection tubes depending on which method of concentration is going to be used. Rinse the empty boiling flask and drying funnel with methylene chloride after the initial transfer to ensure the qualitative transfer of the extract.
- 8.1.3. The KD concentrator apparatus consist of a 500ml KD flask and a 10 or 15ml concentrator tube. The concentrator tube is attached to the flask by placing it on and then giving a gentle twist. The concentrator tube should be on snug. Place a blue Keck clamp to ensure the tube will not fall off. Continue with **Section 8.2.1.**
- 8.1.4. The Caliper TurboVap 200mL collection tubes are placed underneath the drying funnel and the extract is collected directly into it. Continue with **Section 8.2.2.**

8.2. Sample Extract Concentration:

8.2.1. Procedure for concentration of sample extracts using **KD apparatus without further Gel Permeation Chromatography (GPC) cleanup.**

- 8.2.1.1. Add 2 boiling chips to the KD flask containing the extraction fluid.
- 8.2.1.2. Attach a three-ball Snyder column to the KD flask and submerge the evaporator into the hot water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.1.2.1. Concentrate the **SV (or TPH/DRO)** extract down to about 1ml.

8.2.1.2.2. Concentrate the **Pest/PCB** extract down to less than 5ml and then add approximately 20ml of hexane through the top of the Snyder column to exchange the solvent. Continue to concentrate the extract to less than 5ml. REPEAT.

Caution: It is important that the extract in the KD apparatus not be allowed to go dry to minimize volatilization of the more volatile target compounds.

- 8.2.1.3. Remove the evaporator from the water bath. Let the evaporator cool for at least 10 minutes, allowing the solvent in the Snyder column to drain back into the KD concentrator tube. Rinse the KD flask with a small volume of methylene chloride.
- 8.2.1.4. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a 20x150mm disposable culture tube. Rinse the lower joint of the KD flask and the concentrator tube with another small amount of methylene chloride to ensure a full transfer of the extract.
- 8.2.1.5. Place the culture tube into the nitrogen blow down bath, set at 30-35°C (recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.
- 8.2.1.6. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize loss of the more volatile target compounds.

8.2.1.7. Concentrate the **SV (or TPH/DRO)** extract to between 0.5 and 0.8ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to

the 1.0ml mark. Transfer the 1.0ml extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

- 8.2.1.8. Concentrate the **Pest/PCB** extract to 5ml. Transfer the extract to a 15ml vial marked at the 10ml mark. Rinse the concentrator tube with 1ml of hexane and add to vial. Rinse the concentrator tube once more. Bring the final extract volume up to 10ml with hexane. Mark the meniscus with a permanent marker.
- 8.2.2. Procedure for concentration of sample extracts using TurboVap (TV) apparatus without Gel Permeation Chromatography (GPC) cleanup:
 - 8.2.2.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C (recommended).
 - 8.2.2.1.1. Concentrate the **Semivolatile** extracts to 0.5 – 0.8mL. Allow the collection tube to stand without nitrogen flow to cool. Using a 1.0 ml syringe pull up the extract and rinse the interior of the collection tube with the extract. Pull up the extract again with the 1.0 ml syringe. Rinse the collection tube with additional methylene chloride and pull up in the syringe. If needed, add additional methylene chloride to bring the extract to 1.0 ml. Transfer the extract to a 2 ml crimp top autosampler vial. Mark the meniscus with a permanent marker.
 - 8.2.2.1.2. Concentrate the **Pesticide/PCB extract** to about 4ml and then add 20ml of hexane. Raise the temperature to 80 °C (recommended), if necessary, to complete the concentration. Concentrate to about 4 ml again, and add 20 ml of hexane. Continue to concentrate the extract as in **Section 8.2.1.5.2**.
 - Caution:** it is important that the extract in the collection tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.
 - 8.2.2.2. The extracts are transferred to the GC or GC/MS lab and documented in the appropriate Extract Transfer log. The extracts are stored in the appropriate refrigerator, at 4°C ± 2 °C until analysis.
- 8.2.3. Procedure for concentrating sample extracts using the **KD apparatus with further Gel Permeation Chromatography (GPC) cleanup**:
 - 8.2.3.1. Add 2 boiling chips to the KD flask containing the extraction fluid.
 - 8.2.3.2. Attach a three-ball Snyder column to the flask and submerge the evaporator into the water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and

after hexane exchange) with the tip of the KD concentrator tube in the water.

- 8.2.3.3. Concentrate the extract down to less than 5ml and remove the evaporator from the water bath.
 - 8.2.3.4. Let the evaporator cool for at least 10 minutes, and allow the solvent in the Snyder column to drain back into the concentrator tube. Rinse the KD flask with a small volume of methylene chloride to ensure a full transfer of the extract.
 - 8.2.3.5. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a disposable culture tube and place the culture tube into the nitrogen blow down bath, with the temperature set at 30-35°C (recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8. Rinse the lower joint of the KD flask and the concentrator tube with a small amount of methylene chloride to ensure a full transfer of the extract.
 - 8.2.3.6. Start a low flow stream of nitrogen across the surface of the extract solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulates inside the tube. Concentrate the samples to 2ml. This is to reduce the concentration of acetone in the Pre-GPC soil extract.
 - 8.2.3.7. Transfer the 2ml extract into a 15ml vial with a line at the 10ml mark. Rinse the concentrator tube with 2ml of methylene chloride and combine the rinsate into the vial. Rinse the concentrator tube one more time with 2ml of methylene chloride and pour into the vial. Bring up to the 10ml mark with methylene chloride.
 - 8.2.3.8. The extract is now ready for GPC cleanup. Refer to SOP 50.0032 for how to perform GPC.
- 8.2.4. Procedure for concentrating sample extracts using the TV apparatus with further Gel Permeation Chromatography (GPC) cleanup:
- 8.2.4.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C (recommended).
 - 8.2.4.2. Concentrate the extract down to about 2-3 ml and remove the collection tube from the nitrogen blow down bath. Allow to cool without nitrogen flow.
 - 8.2.4.3. Pipet or carefully pour the extract into a 15mL vial marked with a line at the 10ml mark. Rinse the concentrator tube with 2mL of methylene chloride and pour into the vial. Rinse the concentrator tube one more time with 2mL

of methylene chloride and pour into the vial. Bring up to the 10mL mark with methylene chloride.

- 8.2.4.4. The extract is now ready for Gel Permeation Chromatography (GPC) cleanup. Refer to SOP 50.0032 for how to perform GPC Method SW3640.
- 8.2.5. Post GPC the extracts require additional concentration steps.
- 8.2.5.1. For **Semivolatile** extracts, 5ml of the original 10 ml extract was used for cleanup and the final extract volume must be 0.5ml in methylene chloride.
- 8.2.5.1.1. Semivolatile extracts (TPH/DRO samples do not undergo GPC Cleanup) are re-concentrated using the KD until approximately 4ml.
- 8.2.5.1.2. The extract is then transferred to 20x150mm disposable culture tubes and placed into the nitrogen blow down bath, set at 30-35°C (recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.
- 8.2.5.1.3. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.
- Caution:** it is important that the extract in the culture tube not be allowed to go dry to minimize loss of the more volatile target compounds.
- 8.2.5.1.4. Concentrate the **SV** extract to a final volume of 0.5ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 0.5ml mark. Transfer the extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.
- 8.2.5.2. For **Pesticide/PCB** extracts, 5ml of the original 10 ml extract was diluted to a 10ml final volume using methylene chloride, and was used for GPC cleanup. The final extract should be solvent exchanged to hexane and concentrated to 5ml.
- 8.2.5.2.1. The post GPC extract is transferred to a TV apparatus with the temperature set to 60 °C (recommended). Concentrate to about 4 ml. Raise the temperature to 80 °C (recommended), if necessary, to complete the concentration. Add 20ml of hexane to exchange the solvent. REPEAT. Continue to concentrate the extract until it has reached 2-3ml. Transfer the extract to a 15mL vial marked at the 5mL mark. Rinse the collection tube with hexane several times to ensure a full transfer. The final volume must be 5ml. Mark the meniscus with a permanent marker.

8.2.5.2.2. The extract is ready for florisil cleanup. Refer to SOP 50.0033 for detailed instructions on florisil cleanup, Method SW3620.

8.2.5.2.3. Other cleanup methods may be employed if needed. Possible procedures may be: Silica Gel Cleanup, Method SW3630 (SOP 50.0034), Acid Cleanup, Method SW3665 (SOP 50.0031) or Sulfur Cleanup, Method SW3660 (SOP 50.0030).

8.2.6. Excess volume of extracts are stored in screw cap vials and labeled with color coded lab tape. The color of the tape records the extract holding time until disposal. See chart in laboratory.

8.3. Sample and Extract Disposal:

All sample extracts are disposed of in accordance with applicable OSHA and state regulations. All sample extracts must be protected from light and stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

8.3.1. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

8.3.2. EPA CLP/SOM sample extracts- All sample extracts are kept until 365 days after delivery of a reconciled, complete data package.

9. Data Reduction and Calculations

Not Applicable

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contains the manufacturer's reference number given in the standard preparation logbook.

10.1. Method Blank – A method blank is a liter of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank

- The Method Blank is prepared in identical fashion as the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a numerical value, which increases with every batch of twenty samples or less.

10.2. Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3. Lab Control Sample (LCS) – A Lab Control Sample is a liter of organic free reagent water that is spiked with all target analytes and the surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2. Procedure for LCS:

An LCS is prepared in identical fashion as the associated samples; in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the corresponding numerical value as the associated method blank.

10.4. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix. For samples that are known to contain target analytes, the laboratory should

perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples, in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

- 11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. The Quality Control Officer will perform periodic and unscheduled reviews. These data consist of the final volume of sample extracts, the volume and lot number of solvents used, and extract transfer dates.
- 11.2 Reporting of the data will include review by the Organic Preparation Laboratory Supervisor of the data listed in **Section 11.1**, time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP is not adhered to:

12.1 Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. A re-

extraction request will be filled out. The re-extracted samples will be labeled with the suffix RE.

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out. The re-extracted samples will be labeled with the suffix RE.

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, IV, or On-line Revisions of 3500 and 3600 series for Organic Extraction, Sample Preparation and Cleanup.

USEPA Statement of Work, Current OLC, OLM and SOM Methods.

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

**Preparation of Soil Samples by Acid Digestion
for ICP /AES Analysis**

by

SW846 Method 3050B

SOP No. 100.0104

Rev. 8

Signature

Date

QA Director:

Shayr B Saulh

3/29/10

Lab Director:

[Signature]

3/29/10

Effective Date:

4/05/10

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.**

STANDARD OPERATING PROCEDURE

for

**Preparation of Soil Samples by Acid Digestion
for ICP /AES Analysis**

by

SW846 Method 3050B

Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) deals with the preparation of soil samples utilizing USEPA SW846 Method SW3050B for analysis by ICP/AES Method 6010. Discussion includes sample digestion and sample concentration technique for the analysis of metals in soil samples.

2. Personnel Qualifications and Responsibilities

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. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date and that it is implemented appropriately. Supervisors/lab managers review logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 1.00 to 2.00 gram soil/solid sample is digested with the addition of acids and hydrogen peroxide for metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been adapted to utilize polyethylene digestion tubes and a final 50mL digestate volume, rather than the 100mL volume noted in the original method. The reagent volumes have been reduced to accommodate the method as well.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Hold time for ICP analysis is 180 days from date collected.

4.2 Samples are stored at 4°C in amber glass jars with Teflon lined caps.

5. Interferences and Potential Problems

Possible sources of contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware.

5.3 Acid dispensers.

5.4 Sample matrix effects: Extreme organic samples.

5.5 Disodium Stannate preservative for peroxide: Do not use this peroxide when analyzing tin in soils unless proven not to contribute significant tin by analysis of associated Method Blanks. If method blanks show significant tin, an alternative peroxide source must be used.

6. Equipment and Apparatus

Equipment used in this preparation method include:

6.1 10% HNO₃ acid bath.

6.2 50 mL graduated polyethylene tubes.

6.3 Watch Glasses.

6.4 Hot plates.

6.5 Centrifuge.

6.6 Balance for weighing out sample (calibrated daily prior to use).

7. Standards and Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test. Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.

- 7.1 Concentrated HNO₃, ACS trace metals grade.
 - 7.1.1 1:1 (v/v) HNO₃, ACS trace metals grade.
- 7.2 Concentrated HCl, ACS trace metals grade.
- 7.3 30% Hydrogen peroxide (H₂O₂), ACS certified.
- 7.4 Spiking solutions, High Purity Standards.
- 7.5 Teflon chips, Chemware Ultra PTFE Boiling Stones.

8. Procedure

See SOP 110.0039 for sub-sampling procedures.

- 8.1 Digestion of soil samples for ICP/AES analysis:
 - 8.1.1 Note in Soil/Solid Sample Preparation Logbook the presence of any artifacts (see **Figure 1**).
 - 8.1.2 Mix the sample thoroughly so that a homogenous representative aliquot can be taken. Weigh an approximate 1.00 to 2.00 gram (wet weight) or 1.00 gram (dry weight) aliquot of the sample to the nearest 0.01g and transfer it to a 50 mL digestion tube.
 - 8.2.1 Add 5 mL of 1:1 (v/v) HNO₃, mix well. Cover the tube with a ribbed watch glass. Heat to 95°C ± 5 °C in graphite holders on hotplates in the hood, and reflux for 10 minutes without boiling.
 - 8.2.2 Remove from hotplate and allow to cool.
 - 8.2.3 Add 2.5mL of conc. HNO₃. Replace on hotplate and reflux for 30 minutes. If brown fumes are generated at this step repeat the addition of 2.5mL conc. HNO₃ with heating steps until no more brown fumes are given off. Do not allow the volume to fall below 5mL.
 - 8.2.4 Allow the sample to cool and add 1mL of DI water and 1.5 mL of 30% H₂O₂. Return the tube to hotplate to start peroxide reaction, making sure no losses occur due to excessive effervescence action. Heat until effervescence subsides and then allow tube to cool

- 8.2.1 Continue adding 30% H₂O₂ in 0.5mL aliquots with warming until minimal effervescence occurs, the appearance of the sample remains unchanged, or 5mL have been added. If the blank or a sample no longer has effervescence, set them aside, do not continue to add H₂O₂. When all are done proceed to next step.

Note: do not add more than a total of 5mL H₂O₂

- 8.2.1 *For ICP/AES samples ONLY:* Add 5mL of concentrated HCl and return to the hotplate and reflux for an additional 15 minutes.
- 8.2.2 Take tube off the hotplate and cool to room temp.
- 8.2.3 Volumize to 50mL with DI Water.
- 8.2.4 Centrifuge sample for approximately 5 minutes at 3000 rpm to settle any insoluble material.
- 8.2.5 Label the tube properly. All digestion information is documented in the Soil/Solid Sample Preparation Logbook (**figure 1**). Transfer the digestates to Inorganic Instrument Lab.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic prep lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1 Preparation Blanks:

A method blank must be run with every discrete batch of samples that is being prepped. A batch of samples cannot exceed 20 samples.

- 10.1.1 The method blank includes all reagents used to prepare samples and is treated as if it were a regular sample. Label appropriately.

10.2 Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch cannot exceed 20.

10.2.1 Measure 1.0 ± 0.01 g acid washed Teflon chips into a 50mL -digestion tube. Spike 455 uL of CV-1 and 45.5 uL each of CV-2 and CV-3 into the tube and digest as described in **Section 8**. Label appropriately. .

10.3 Matrix Spikes and Duplicate Samples:

Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 1.00-2.00 g (wet weight) or 1 gram (dry weight) of sample (the same sample).

10.3.1 Matrix Spikes:

- To one aliquot add three spiking standards containing all target analytes; High Purity Standard CLP-CV-1 at 455 uL; CLP-CV-2 and CLP-CV-3 at 45.5 uL each. Label this sample with an “MS” suffix, and digest as described in **Section 8**.

10.3.2 Duplicates:

- Label the tube designated for the sample duplicate with a “D” suffix and digest as described in **Section 8**.

10.4 Standard Preparation

All standards made from a primary standard expire on or before the primary standard's expiration date.

10.5 Digestion Tubes and Pipettes

10.5.1 All lots of digestion tubes must be tested for conformance. A group of 10 tubes are measured for volume by weight. The weights are recorded and the average weight is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

10.5.2 Pipettes: Two sizes of pipettes are used in the method; 0 to 1000 μ L and 0 to 100 μ L. Five replicate weights are recorded. The average of the five replicates is calculated. See SOP 80.0030 Labware Volume Verification for acceptance criteria.

11. Data Validation and Reporting

Data (logbook entries) generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the department manager.

12. Corrective Action Procedures

All corrective action will stem from the analytical results in the Metals Laboratory. See the specific SOP for details on QC requirements.

12.1 If any method blank shows contamination, associated samples are scheduled for reprep by the department supervisor or manager.

12.2 If any spike recovery or duplicate RPD is outside of control limits, samples are scheduled for reprep by the department supervisor or manager.

12.3 If a Spike sample was not spiked, it will be re-prepped and re-analyzed with the appropriate sample and duplicate.

13. Health and Safety

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

U. S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, SW-846, Update III, Revision 2, December 1996, Method 3050B.

Attachments:

Figure 1: Soil/Solid Sample Preparation Logbook

Figure 1

Soil/Solid Sample Preparation Logbook

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

**Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
(GC/MS) Analysis by SW846 Method 8260C**

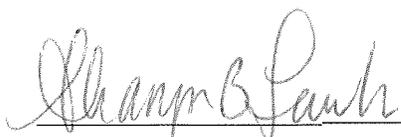
SOP No. 90.0012

Rev. 11

Signature

Date

QA Director:



3/15/10

Lab Director:



3/19/10

Effective Date:

3/26/10

**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

**Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
(GC/MS) Analysis by SW846 Method 8260C**

Rev. 11

1. Scope and Application

This SOP describes the analysis of volatile organic compounds in aqueous and soil samples using gas chromatography/mass spectrometry (GC/MS). The SOP covers the analyses according to protocols discussed in SW846 Update 8/2006 of Method 8260C. A modified version of Method 8260C for a low level aqueous analysis using a 25 mL aliquot is included as **Section 16**. This SOP meets all of the requirements specified in the method. To further familiarize oneself with the procedures, the analyst is encouraged to consult the following instrument manual:

- Hewlett Packard EnviroQuant GC/MS Manual

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol or unusual occurrences noted during analyses. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve reported results. The **Laboratory Director** and/or senior management evaluate laboratory reports for reasonableness of the results and sign the reports. The **QA Director** reviews the QC system and quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

- 3.1 The volatile compounds are introduced into the GC/MS by purge-and-trap system. Analytes are extracted from the sample by bubbling with helium. The analytes are trapped from the helium stream on an adsorbent trap. The analytes are desorbed at high temperature directly onto a narrow-bore capillary column after been split at 1:50 ratio via an EPC controlled injector for analysis. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer.
- 3.2 Analytes eluted from the capillary column are introduced into the mass spectrometer by direct interface to the ion source. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (primary) ion relative to an internal standard using a five-point calibration curve.
- 3.3 A list of abbreviations used in this SOP is included in **Table 1**.
- 3.4 The list of compounds to be analyzed and reported may vary from project to project. The SW-846 method contains several different lists of analytes, so there is no "official" EPA list of Method 8260 compounds. Mitkem typically analyzes samples for and reports a fairly extensive list of analytes. Certain projects also may have additional extra compounds not on the normal Mitkem list. Alternatively certain projects may have a shorter list of compounds than the normal Mitkem list. These project-specific lists of analytes are specified by the client through discussion with the Mitkem Project Manager who discusses the list with the Laboratory Supervisor. The lists are managed in the lab by the use of "sublists" in the Target data reduction and reporting software. In addition, when utilizing the LIMS system, the sublist can be viewed using the SEL list option. SEL refers to the select list of target analytes requested by the client. It is used when this list differs from the "routine" analyte list. A list of the routine 8260 analytes used by the laboratory is shown in **Attachment 1**. Refer to the LIMS Test Information category/Test option/ limits of the test code, for the most current MDL values. Those listed in **Attachment 1** may not be the most up to date.
 - 3.4.1 Several options exist for reporting extra compounds not on the normal Mitkem list. The ideal approach includes purchasing a calibration standard solution and a second source calibration check solution, determination of method detection limit from 7 or more replicate analyses and addition of the compound to initial and continuing calibrations and laboratory control sample analyses. (See Mitkem SOP No. 80.0005 for details on determination of the MDL). Depending on the needs of the client, alternative approaches may be appropriate, including single point calibration or searching for the compound as a Tentatively Identified Compound using the Target software's library search routines. The approach taken must be discussed with the client prior to

analyses, and if needed, sufficient documentation is included in the analysis report to enable validating the data. The analyst will be instructed by the lab supervisor as to what documentation is needed and what is required to be sent to the data reporting area for inclusion in the final report.

- 3.4.2 The Quality Control requirements contained in this SOP apply to the specific list of analytes being reported. SOP criteria are to be evaluated for all project target analytes. While QC issues with non-project target analytes should be investigated, they are not critical if that compound is not being reported. Mitkem's calibration standards and LCS/MS solutions may contain compounds not being reported for a particular project.

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For volatile organic compound analysis by method 8260, water samples are collected in 40-milliliter (mL) glass vials, typically preserved with HCl. Solid samples may be collected in glass containers, EnCore™ samplers, pre-weighed 40-mL vials preserved by 5 mL of DI water to be frozen upon receipt or similar pre-weighed vials with sodium bisulfate solution for low-level analysis or preserved by 5 mL methanol for medium-level analysis. According to Method 5035, the low-level soil samples shipped in EnCore samplers need to be extruded into a pre-weighed DI water/stir bar vial or similar vial with sodium bisulfate solution as soon as received in the lab (within 48 hours of collection). If needed, the soil samples received in EnCore samplers may be preserved by storage in a dedicated freezer until prior to the analysis. This freezer only contains samples and must not contain any analytical standards. ASTM method D6418-04 includes documentation of the ability of EnCore devices to contain volatile compounds without significant loss when frozen for up to 14 days. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples. Typical sample submittals are listed below:

- “normal aqueous samples: 2 X 40ml vials, HCl Preserved
- “low-level” aqueous samples: 3 X 40ml vials, HCl Preserved
- preserved soil samples: 2 X DI water/freeze preserved or sodium bisulfate preserved plus
1 X methanol preserved plus a four-ounce jar for percent solids determination

Other sample submittals may be suitable for analysis depending on the needs of the specific project. The Project Manager, Supervisor or client should be contacted to determine the suitability of sample containers to meet SOP and project objectives.

- 4.2 All aqueous samples and soil samples received in glass jars are stored in the VOA lab at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ until analyzed. The soil samples received in 40mL vials preserved either by sodium bisulfate or methanol are also stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in the VOA lab. Soil samples received in pre-weighed 5mL DI water vials are stored in a freezer at down to -20°C . For soil samples received in the EnCore type of device (typically in silver pouches), these are extruded into pre-weighed vials containing a stir bar and 5mL of DI water, then re-weigh the vials to obtain the final sample weights. The vials are to be placed into a freezer.
- 4.3 Storage areas used for samples for volatile organic analysis must be free of potential contaminants. To document storage conditions a storage blank consisting of a 40ml vial of organic-free water is placed in every refrigerator used to store VOC samples on a weekly basis. Storage Blanks (refrigerator and freezer where applicable) are logged in to the LIMS system and tracked using the reporting feature. These will be analyzed a weekly basis. When a storage blank is removed for analysis, another blank will be placed in the refrigerator such that there will be a blank in each refrigerator on a 24/7 basis. Highly contaminated samples are stored in a specially marked refrigerator in the VOA lab.
- 4.4 Sample holding time for volatile organic compound analysis by method 8260 are 14 days from the day of sample collection for both preserved aqueous and soil samples. The holding time for non-preserved aqueous samples is 7 days from the day of sample collection. Samples will be disposed after a minimum of 30 days after the submission of a complete data package.

5. Interference and Potential Problems

- 5.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non PTFE thread sealant, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting value result in what the laboratory feels is a false positive result for a sample due to laboratory background contamination; the laboratory should fully explain this in text accompanying the uncorrected data. Compounds detected in method blanks and also detected in samples from the same batch are qualified with a "B" flag on data report forms, and listed on the data review checklist. The definition of the "B" qualifier is included in the data report.
- 5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the purging apparatus and sample syringes must be rinsed with at least two portions of organic-free

reagent water between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of one or more blanks to check for cross-contamination.

- 5.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device in the sample flow path including purging vessel, tubing, or sample valves may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is helpful to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by analysis of an extra aliquot at a dilution beyond the 12 hour instrument tune time or comparison to any available previous results for the sample.
- 5.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination. It is important for analysts to keep this in mind if they enter the organic preparation laboratory and plan to return to the volatiles lab. Their clothing may be a source of contamination in the volatiles lab.
- 5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water or other matrix and carried through the sampling, handling, and storage protocols serve as a check on such contamination. Trip blanks are typically sent with each shipment of VOA vials and the client is advised to have them analyzed. The client is also advised to prepare field blanks to be sent to the lab if appropriate for their sampling plan.
- 5.6 Use of sensitive mass spectrometers or larger sample sizes to achieve lower detection levels will increase the potential to detect laboratory contaminants as interference.
- 5.7 Direct injection (for BFB) – Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in case of extreme contamination.

6. Equipment and Apparatus

6.1 Equipment:

- 6.1.1 There are four GC/MS instruments in the Volatile Organic Analysis Lab. There are V1, V2, V5 and V6. The GC/MS systems have similar configurations as follows: Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V1); Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V2); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V5); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5973 mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V6). The laboratory maintains flexibility to interface various purge and traps to various autosamplers and subsequently to various GC or GC/MS systems. This flexibility is an important tool in trouble-shooting system problems or to minimize the impact of instrument down-time. EnviroQuant Software is used to handle data acquisition. Data files are copied and transferred to the company file server via the computer network. Actual data quantitation and analyses are performed by the analyst using Target chromatographic software (Thru-Put systems, Inc.).
- 6.1.2 A 30m x 0.25mm id DB-624 capillary column is used for all three GCs. The GC is directly interfaced to the MS. The GC injector operates under split mode (about 50:1) at all times.
- 6.1.3 OI 4552 autosamplers are fitted with heat function for low level soil analysis (V2, V5 and V1).
- 6.1.4 High purity helium (99.999%) is used both as GC carrier gas and Purge and Trap purge gas
- 6.1.5 The instruments scan from amu 35 to 300 at EM voltage similar to those of the tunes.
- 6.1.6 The BFB data acquisition method is V1TUNE, V2TUNE, V5TUNE and V6TUNE for each of the four instruments.
- 6.1.7 The data acquisition methods for unheated purge (for aqueous and medium level soil samples) are V1VOA, V2VOA, V5VOA and V6VOA. The data acquisition methods for heated purge (for low-level soil samples) are named similarly.

6.1.8 The purge and trap systems operating conditions are as follows:

Aqueous (including both 5mL and 25mL samples) and medium soil samples

Purge	11 min at ambient temperature
Dry Purge	2 min
Desorb*	2 min at 190 °C
Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)
Transfer Line Temp.	125 °C

Low level soil samples

Preheat	2 min
Purge	11 min at 40 °C
Dry Purge	2 min
Desorb*	2 min at 190 °C
Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)
Transfer Line Temp.	125 °C

* Desorb time and Bake times may be varied to optimize the instrument performance.

6.1.9 Instrument operating conditions are as follows:

General Gas Chromatography Conditions

For V1 and V2:

Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	10 °C/min to 120 °C, then 15 °C/min to 240 °C
Final Time	2 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

For V5 and V6:

Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	12 °C/min to 200 °C, then 20 °C/min to 240 °C
Final Time	2.7 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

General Mass Spectrometry Conditions

For V1 and V2:

Mass Range	35-300 amu
Scan Speed	1.6 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune

For V5 and V6:

Mass Range	35-300 amu
Scan Speed	0.97 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune

6.1.10 Balance

A top-loading balance capable of weighing 200.0 ± 0.1 g.

6.2 Preventative Maintenance - The Purge and Trap GC/MS are maintained according to the manufacturers' recommendations. The lab analyst performs preventive maintenance as discussed below:

6.2.1 The Purge and Trap spargers are rinsed and cleaned automatically by autosampler between each analysis. This cleaning procedure is sufficient for most sample analyses. Some samples exhibited target compounds or TIC at unusual high concentrations result in contaminating the Purge and Trap system that may require additional cleaning and baking out. Under the circumstances, any part on the flow path directly contacting with sample including transfer line, valves and sparger may need to be back-flushed with VOC-free water, then with methanol, and extra baking. Analyst performs this extensive cleaning procedure should record them into the LIMS maintenance logbook (see **Section 6.2.9**). The system must be demonstrated to be contaminant free by analyzing instrument blank before reuse for sample analysis. The trap will be replaced if tailing peak response and loss of gaseous compounds that can not be related to standard solution problems are observed.

- 6.2.2 The GC septum will be replaced monthly (this is done rarely as the septum is only penetrated whenever BFB tuning compound is analyzed).
 - 6.2.3 The maintenance of GC injection liner will be performed as needed. If necessary, the liner will be replaced.
 - 6.2.4 If needed, the analytical column will be replaced; this is usually indicated by excessive peak tailing and/or repeated failures of initial or continuing calibration verifications to meet SOP criteria.
 - 6.2.5 The ion source will be cleaned when the system drifts out of BFB tune and/or repeated failure of initial and/or continuing calibrations to meet SOP-specified criteria.
 - 6.2.6 If there is a second blown filament, the ion source will be vented to install two new filaments. Whenever the ion source is opened for maintenance, the analyst should make sure two good filaments are in place, or replace any filament blown since the last maintenance. This will minimize the times when both filaments are blown.
 - 6.2.7 The pump oil will be replaced as needed.
 - 6.2.8 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the manufacturer of the problem to schedule on-site diagnosis and repair.
 - 6.2.9 All non-routine preventive and corrective maintenance shall be documented in the Instrument Maintenance log located in the LIMS system. This can be accessed using the category Analytical and option Instruments in the LIMS menus. All analysts have access to this function in LIMS. If help is needed, ask the Lab Supervisor for assistance.
- 6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD and 5973 MSD hardware manuals.
- 6.4 Glassware:
- 6.4.1 Class "A" volumetric flasks: 10 mL and 100 mL.
 - 6.4.2 Syringes: 2 uL, 5 uL, 10 uL, 25 uL, 50 uL, 100 uL, 500 uL, 2.5 mL, 5 mL, and 25 mL.
 - 6.4.3 Syringe valve - Two-way, with Luer ends (three each) if applicable to the purging device.

7. Reagents and Standards

- 7.1 Analyte-Free Reagent Water (also referred-to as VOC-free water or DI water elsewhere in this SOP) - prepared by eluting tap water through a column of activated charcoal granules to remove traces of volatile organic compounds, or the Whirlpool Water Filter system.
- 7.2 Purge and trap grade methanol - from Fisher Scientific *or equivalent* quality of solvent from other vender will be used for standard preparation. Each new batch of solvent is checked by analyzing a 200 μ L aliquot of the methanol in a 5mL aliquot of pure water, or 1.6mL per 40ml vial of pure water. The new batch is acceptable if the analysis does not detect any contaminants that interfere with the measurement of target analyte compounds, or contain unacceptable levels of non-target compounds. While the criteria for method blank evaluation can be used for guidance, stricter criteria will be beneficial as potential contamination from various sources may add-up to impact the analysis.
- 7.3 The standards used for this SOP are discussed below. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.* Stock solutions for calibration standards are:

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Ultra	DWM-589N	2000 ug/mL
Gas Mix	Restek	30042	2000 ug/mL
Ketone Mix	Restek	30006	5000 ug/mL
Additional Mix	Ultra	CUS-6268	400-8000 ug/mL
Internal Standards	Restek	30241	2500 ug/mL
Surrogates	Restek	30240	2500 ug/mL

7.3.1 All of the primary standards are labeled as VPymmddX,

where: VP = Volatile **P**rimary standard
 yymmdd = date the standard is received
 X = the order that the standard is logged into the Log Book on that date,
 in increasing alphabetical order

7.3.2 All unopened ampulated primary standards will be replaced either by following the expiration instructions from the manufacturer, or after two years from the date received if no expiration date was provided. For an opened stock standard ampule, replace after **6 months** from the date it was opened or sooner if the standards have degraded or evaporated.

7.3.3 Standards are stored separate from samples, at a temperature of $\leq 6^{\circ}\text{C}$ or as recommended by the vendor. Mitkem stores standards in a separate freezer which is maintained between -10 and -20°C .

7.4 Stock Solution (independent “second” source, *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to NIST reference materials.*) for ICV:

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Accu Standard	M-502A-R	200 ug/mL
Gas Mix	Accu Standard	M-502B	200 ug/mL
Additional Mix	Accu Standard	M-8260-ADD	200 ug/mL

7.4.1 The labeling and storage for all ICV primary standards should be handled following the similar procedures for primary calibration standards listed above in **Section 7.3.1 through 7.3.3**.

7.5 Working Standard solutions:

7.5.1 Working standard solution for calibration mix: place 2520 μL of methanol into a 4mL vial fitted with Teflon septum. Transfer 200 μL of 8260 Standard Mix (Cat. No. DWM589N), 200 μL of Gas Mix (Cat. No. 30042), 80 μL of Ketone Mix (Cat. No. 30006) and 1000 μL of Additional Mix (Cat. No. CUS-6268) into the vial to make a solution of all analytes at 100 ug/mL.

Note: using the syringe, add the standards below the surface and into the methanol by pushing the syringe plunger smoothly to the end. Make sure all of the standard mix is transferred into the methanol before the syringe is removed.

Gently invert the 4mL vial several times to ensure proper mixing. Repeat this process for all mixtures. This working standard is then transferred into four 1 mL vials with mininert valve. Keep all four vials in freezer at -10 to -20°C . Only take one vial out and use it for calibration and QC samples. A new vial should be taken out and used weekly, or as needed based on standard degradation.

7.5.2 Working standard solution for ICV: place 700 μL of methanol into a 1mL vial fitted with Teflon septum. Transfer 100 μL each of Non-Gaseous standards (M-502A-R), 8260 Additional standards (M-8260-ADD) and Gaseous standards (M-502B) into the vial to make a solution of all analytes at 100 ug/mL.

7.5.3 Surrogate Standard solution (SS): four surrogate compounds are used for analysis: dibromofluoromethane, 1,2-dichloromethane-d4, toluene-d8, and bromofluorobenzene. The working standard is prepared by transferring 160 uL of the stock surrogate standard solution (Cat. No. 30240) into a 4 mL vial with 3840 uL of methanol to make a solution at 100 ug/mL.

7.5.4 Internal Standard solution (IS): three internal standards used for analysis: fluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene. The working standard solution is prepared by transferring 160 uL of the stock internal standards (Cat. No. 30241) into a 4 mL vial with 3840 uL of methanol to make a solution at 100 ug/mL.

7.5.5 Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 400 uL each of the IS stock solution (Cat. No. 30241) and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3200 uL of methanol to make a 250 ug/mL solution. Once prepared this solution is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 5 mL purge volume, this yields a concentration of 50 ug/L or ug/Kg.

7.5.6 All of the working standards are labeled as VWyymmddX,

where: VW = volatile working standard
yymmdd = date the standard is prepared
X = the order that the standard is logged into the Log Book on that date,
in increasing alphabetical order

See **Table 2** for details on making working standard solutions. Working Standards are good for **one month**. All standards made from a primary standard must not exceed the primary standard's expiration date.

7.6 All of the standard information is recorded in the LIMS standard/spike Logbook upon receiving or preparation (see Figures 1 and 2). All vials containing working standards must be labeled according to the current version of SOP 80.0001 Standard Preparation, Equivalency and Traceability. Be sure the vial label is not worn or difficult to read. Any vial whose label becomes worn or difficult to read should be re-labeled.

8. Procedure

8.1 Tuning:

8.1.1 The GC/MS must be tuned to meet 4-bromofluorobenzene (BFB) criteria every 12 hours when standards, blanks or samples are to be analyzed.

All of the analysis information is to be recorded in the Instrument Run Logbook (**Figure 3**). The logbook is issued by the QA officer and will be returned back to the QA officer for archiving when all pages are used.

8.1.1.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.1.1.5** to perform the tune analysis.

Inject 2 μL of the working tune standard (50 ng) directly into the GC/MS through the septum injection port using a 10 μL syringe. Alternatively, the BFB can be introduced through the purge and trap system, as a sample is.

A typical BFB chromatogram is shown in **Attachment 2**.

8.1.1.2 Acceptance criteria for tune - The mass spectrum of BFB must be acquired across the peak. The primary mean for evaluating ion abundance is averaging three scans: the peak apex scan and the scans immediately preceding and following the apex. One of following alternates may be used to evaluate the tune: averaging the entire BFB peak, the single scan of apex, the single scan before apex, or the single scan after apex. Background subtraction is required and accomplished by subtracting a single scan no more than 20 scans prior to the beginning of the elution of BFB. It is important that the analyst does not selectively add or subtract scans to meet the tune criteria.

A typical mass spectrum and mass spectral listing of the tune in listed in **Attachment 3**.

The acceptance criteria are as follows:

<u>Mass</u>	<u>Ion Abundance</u>
50	15.0 - 40.0% of mass 95
75	30.0% - 60.0% of mass 95
95	base peak, 100%
96	5.0 - 9.0% of mass 95
173	< 2.0% of mass 174
174	>50.0 % of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101.0% of mass 174
177	5.0 - 9.0% of mass 176

Once the mass spectrometer passes the BFB tune, all subsequent blanks and standards that are analyzed within the 12-hour shift must be analyzed using identical mass spectrometer instrument conditions.

8.2 Initial Calibration - Initial Calibration is performed after the instrument passes the tune requirements. Initial Calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial Calibration will also be performed if Continuing Calibration analyses do not meet QA/QC criteria.

8.2.1 Five calibration standard solutions are required for all target compounds. Standard concentrations of 5, 20, 50, 100, 200 µg/L (or in µg/kg for soil samples) for typical 5 mL or 5 g sample analyses; 0.5, 4, 10, 20, 40 µg/L for 25 mL aqueous sample analyses are required. (See Section 16 for more details on low level calibration) The lowest standard concentration is typically at or below the reporting limit for the analysis. This is the level closest to the method detection limit (MDL). There may be project-specific requests to calibrate the instrument to the 1 ug/L level using a 5ml purge. This procedure may involve the addition of a sixth level to the initial calibration at the 1 ug/L concentration, or replacement of one of the other concentrations to maintain a 5 level calibration. This is determined by the requirements of the specific project through discussions between the Project Manager and Supervisor. There also may be occasional requests to report results to a limit below the lowest initial standard concentration. These must be documented and discussed in the report narrative. Any request for non-routine calibration should be discussed with the laboratory Supervisor and Project Manager to insure the resulting data meets project and method requirements and the procedures used and the quality of the data are fully documented.

DoD– the ICAL range shall consist of a minimum of 5 contiguous calibration points for organics, for all target analytes and surrogates reported. The low-level standard must be less than or equal to the reporting limit.

Several state and government programs have specific QA/QC Requirements and Performance Standards for the Initial Calibration. Refer to the individual state/government documents for more details. In particular, Dept. of Defense requires the evaluation of SPCC/CCC compounds in both the ICAL and CCV. See Attachment 5 for criteria.

Low level (25 mL) Calibration is documented in **Section 16**.

The calibrations for aqueous and medium level soil samples are performed at ambient temperature purge. The calibrations for low-level soil samples are performed at the same temperature used for sample analysis, using heated (40°C) purge condition.

8.2.2 Initial Calibration standards are made-up as follows:

Initial calibration standards are prepared by adding working standards into 40mL organic-free water. In aqueous analysis, 5mLs of each 40mL solution

are transferred into the purge chamber by the auto-samplers. In soil analysis, 5mL of each 40mL solution are transferred into vials with Teflon caps manually using gas tight syringes and these vials are heated and purged. The IS/SS may be added automatically by the autosampler.

DoD ICALs require surrogates to be added manually (to achieve multiple level calibration) according to **Table 2**.

Calculation for Initial Calibration - A typical chromatogram of a 50µg/L standard followed by the Quantitation Report is shown in the attachments to this document.

From the 5 level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A_x = area of the characteristic ion for the target compound to be measured

A_{is} = area of the characteristic ion for the associated internal standard

C_{is} = concentration of the internal standard

C_x = concentration of the compound to be measured

When using the DB624 column, note the following when determining the RRF:

- Cis- and trans-1, 2-dichloroethenes are separately calibrated.
- O-Xylene is calibrated by itself; the m- and p-isomers are summed together.

The mean relative response factor is determined by averaging the 5 level values.

The % Relative Standard Deviation (%RSD) of the RRF is also calculated using:

$$\% RSD = \frac{SD}{Mean} \times 100$$

where: SD = Standard Deviation, and

$$SD = \sqrt{\frac{(X_i - X)^2}{n-1}}$$

where: X_i = each individual value used to calculate the mean

\bar{X} = the mean of n values

n = the total number of values = 5

8.2.3 Initial Calibration acceptance criteria for SW-846 8260C protocol is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of -50% to $+100\%$ of the mean area response of the internal standard in all of the calibration levels.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments.
- A minimum RRF is suggested, see **Table 3**.
- No quantitation ion may saturate the detector.
- Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the initial calibration is deemed acceptable if the following conditions are met (in order of preference):

- (1) Ten percent (10%) of the compounds are allowed to be greater than 20% RSD with a maximum of 50% RSD. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are not allowed as one of the outliers. Initial Calibrations with over 10% of the compounds above the 20%RSD may be used for screening purposes.
- (2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)

m = slope of the line

x = concentration of the calibration standard

b = intercept

It is important that the origin (0, 0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$.

- (3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)

a and b = slope of the curve

x = concentration of the calibration standard

c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0, 0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the “goodness of fit” is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be ≥ 0.99 .

- 8.2.4 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 50 ppb standard prepared in **Section 7.4**. The standard ID is documented in the run log. The acceptance criteria are as follows:

For routine SW 8260 analyses, the calculated value of the analyte in the ICV should be 70 – 130% of the expected value (35 – 65 ng/uL).

For DoD analyses, the calculated value of the analyte in the ICV should be 80-120% of the expected value (40-60 ng/uL), with no allowance for poor performing compounds.

If the above criteria are not met, the analyst has to evaluate the integrity of the primary and second source standards. First, reanalyze the ICV. Preparation and

analysis of a new initial calibration may be required; however failure to meet the control limits does not in itself negate the Initial Calibration validity. Certain compounds may not meet the criteria under the best of circumstances. Some compounds will require a wider recovery limit. In some cases, the analysis of samples may be used for screening purposes when the ICV fails.

- 8.2.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action included preparing fresh standards, source cleaning, reconditioning or changing the trap. Document the actions and resolution in the LIMS maintenance log.
 - 8.2.6 Initial calibration acceptance criteria must be met before any sample, blanks or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of an initial calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 20% of the non-CCC analytes (calibration check compounds (CCC) are: vinyl chloride, 1,1-dichloroethene, chloroform, ethyl benzene, toluene, and 1,2-dichloropropane) to have %RSD > 30 or $r < 0.99$. This situation is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.
 - 8.2.7 If necessary, the reference spectra in Target are updated from mid-point calibration (50 ppb standard), or from the continuing calibration standard.
 - 8.2.8 Upon the successful completion of the initial calibration, the raw data are arranged in increasing concentration levels together with BFB tune. Raw data include chromatograms and quantitation reports plus any documentation of manual integrations. Refer to SOP No. 110.0008 for details on the need for and documentation of manual integration. A copy of the initial calibration summary listing the RRF and %RSD of each target analyte is also included. These raw data are to be filed separately for the four instruments.
 - 8.2.9 Initial calibration data must be archived in the company's organic analysis calibration (OCAL) database. The information in **Section 8.2.8** is brought to the Data Reporting area and left in the tray for filing OCAL data. The Data Reporting department will scan the calibration printouts into the optical filing database for long-term archiving. This may be done at anytime after the ICAL is deemed acceptable.
- 8.3 Continuing Calibration (CCV) - Continuing Calibration using standards containing all the target compounds at 50 µg/L (or 50 µg/kg) are performed every time samples are

analyzed to ensure that the GC/MS continues to meet instrument sensitivity and linearity requirements.

8.3.1 Frequency of Continuing Calibration - The Continuing Calibration must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the Initial Calibration, samples may be analyzed using the mid-point ICAL standard as the continuing calibration verification. The Continuing Calibration is required whenever blanks, LCS and samples are analyzed.

8.3.2 Procedure for performing Continuing Calibration - The Continuing Calibration is performed at 50 µg/L (µg/kg) injection. The IS/SS are added automatically by the autosampler. Calculate the % difference between the Continuing Calibration RRF and those from the most recent Initial Calibration.

The % difference is determined as follows:

$$\% \text{ Difference} = \frac{\text{RRF}_c - \text{RRF}_i}{\text{RRF}_i} \times 100$$

where: RRF_c = relative response factor from continuing calibration

RRF_i = mean relative response factor from the most recent initial calibration that meets acceptance criteria.

Use % Drift when using least squares or non-linear calibration.

$$\% \text{ Drift} = \frac{\text{Conc}_c - \text{Conc}_t}{\text{Conc}_t} \times 100$$

where: Conc_c = concentration obtained from continuing calibration

Conc_t = theoretical concentration of standard

8.3.3 Continuing Calibration acceptance criteria:

- The RRF for each compound should be greater than those listed in **Table 3**.
- Twenty percent (20%) of the compounds are allowed to be greater than 20 %D, with a maximum of 50 %D. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are not allowed as one of the outliers.
- The area response for the internal standards must be within the inclusive range of -50% to +100% of the area response of the internal standards in the mid-point ICAL standard level.

- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (50 ug/mL) of the associated initial calibration.
- No quantitation ion may saturate the detector.

Several states have specific QA/QC Requirements and Performance Standards for the Continuing Calibration. Refer to the individual state documents for more details.

- 8.3.4 Corrective Action for Continuing Calibration - Investigate the calibration to confirm that calculations have been performed correctly and that all integrations are correct. Depending on which compound(s) fail(s) the criteria, corrective action includes preparing fresh standards, source cleaning, reconditioning or changing trap. Repeated failure to pass continuing calibration may necessitate performing new initial calibration. See **Attachment 8** for specific QC criteria and corrective action.

Note—the following symptoms and corrective actions commonly occur in this analysis. If the gaseous compounds are low, this typically indicates too high purge flow rate, “blowing” these compounds through the trap. The gaseous compounds are also more sensitive to small leaks in the system between the purging chamber and the injection port. If the higher boiling point compounds are too low, this typically indicates too low purge flow, or too low desorb temperature. A cold spot in the transfer line could also cause loss of the higher boiling compounds. If the brominated compounds or 1,1,2,2-tetrachloroethane are low, this typically indicates active sites in the system causing break-down of these compounds. If methylene chloride or acetone are too high or too low, this typically indicates contaminated “blank” water used to make the CCV or possibly in the ICAL (CCV too low).

- 8.3.5 Continuing calibration acceptance criteria must be met before any samples or QC is to be analyzed. There may be circumstances where project-specific criteria allow the use of a continuing calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 10% of the non-CCC analytes to have %D > 30. This is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

- 8.4 Sample Analysis - Samples are allowed to warm to ambient temperature before analysis.

- 8.4.1 Aqueous (method SW5030) - Samples are analyzed in 5mL or 25mL aliquots depending on desired reporting limits or by project specification. The sample is entered into the instrument run log, the bottle number documented, and the vial is placed in a location in the autosampler tray. Immediately prior to analysis, an aliquot of the sample is withdrawn from the VOA vial by the autosampler using a syringe. An aliquot of the combined internal standard/system monitoring standards (IS/SS) are added and the sample aliquot is transferred to the purge-and-trap sparger and injected into the sparger vessel. The sample is ready for analysis.
- 8.4.2 Soil samples are analyzed using heated purge for low level analysis and methanol extraction approach for medium level analysis.
- 8.4.2.1 Low level soil analysis (method SW5035) – Samples are received preserved in DI water or sodium bisulfate (NaHSO₄) solution. The vial also contains a small Teflon-coated stir bar. The “empty” vial/preservative solution/stir bar is weighed prior to shipment to the client/field. The vial is reweighed prior to analysis and the sample weight is determined by the difference in weight. The weight is recorded in the appropriate log book, and the sample and its jar number are logged into the instrument run log book. The vial is allowed to warm to room temperature and loaded into the autosampler. Prior to analysis the autosampler places the vial in a temperature controlled heating block to equilibrate to the analysis temperature 40 °C. For low level soil analysis, the instrument calibration and all QC analyses are to be performed at the same temperature (40 °C) as the sample analyses.
- 8.4.2.2 EnCore Samples – Samples collected into self-contained EnCore (or similar) devices are often collected. Samples are extruded from the EnCore into preservative solution (typically two EnCores into DI water and one into methanol. The aliquots are then analyzed by the low level soil or medium level soil procedure as appropriate.
- 8.4.2.3 Unpreserved Soil Samples – Samples for Method 8260 should be preserved per Method 5035. If soil samples are received unpreserved, but per discussion with the client they are still to be analyzed, the following procedure is used. Approximately 5.0-5.5g soil is weighed into a pre-weighed vial containing DI water and a stir bar. This should be done as soon as possible following sample receipt. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. The soil must be below the surface of the DI water in the vial. The sample is then batched up at the autosampler per the procedures listed under **section 8.4.2.1** above.

- 8.4.2.4 Medium level soil analysis – using field-preserved methanol sample aliquots. The customer collects approximately 5 g of soil sample into a pre-weighed 40mL vial containing 5mL methanol. At the laboratory the sample is weighed again to determine the soil weight by difference. A portion of the methanol extract is transferred into a 40 mL vial for analysis. The typical maximum methanol-water ratio is 100 μ L of the methanol extract added to a total volume 5 mL sample, or 800 μ L to a 40 mL vial. The prepared sample is analyzed using the aqueous sample procedure, using the water calibration to quantitate the medium level analysis.
- 8.4.2.5 Medium level soil analysis – if no field-preserved aliquot is submitted for analysis. Weigh 5.0-5.5g of soil sample into a 15 mL vial, and then quickly add 5mL of methanol. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. Cap and shake for 2 minutes. After phase separation, the methanol extract is transferred into a 4mL vial with no headspace for storage. When the extract is ready for analysis, up to 100 μ L of the methanol extract is added to a 5 mL aliquot of analyte free water, or 800 μ L to a 40 mL vial of DI water. Use the water calibration to quantitate the medium level analysis.
- 8.4.3 Sample Dilution - Sample dilution is performed to ensure that all of the target analytes are determined within the instrument calibration range. Based on the concentration determined in the initial sample analysis, if needed, the analyst will determine the dilution factor required to perform the diluted analysis such that the target compounds will be determined at or above the mid-point calibration. It is important to note that due to over-saturation (column or detector), the target compounds that were determined to exceed the calibration range are usually underestimated (detected concentration lower than actual) in the initial run.
- 8.4.3.1 Low level aqueous sample – Dilutions for aqueous samples are prepared in an appropriate sized volumetric flask. Approximately 40mL of analyte-free water is added to the volumetric flask. The proper volume of sample is measured in a gas tight syringe. The unopened sample vial is used for the dilution. The sample is withdrawn by removing the cap; the septum seal is not punctured. The measured amount of sample is slowly injected into the volumetric flask below the surface of the analyte-free water. The flask is filled to the lip with analyte free water and closed. The diluted sample is then transferred to a 40 mL VOA vial for analysis. The amount of sample used to prepare the dilution must be noted on the logbook along with the final dilution factor to allow for double-checking of dilution calculations. Any

secondary dilution used must be clearly described in the logbook. If more space is necessary, the back of the logbook page is to be used, with a note on the front of the page to refer to the back of the page. If an unopened vial is not available for dilution analysis, the situation must be discussed with the Supervisor and Project Manager prior to using a previously opened vial. If a previously opened vial is approved for use, this must be noted on the run log book and on the data review checklist submitted with the data for review to allow discussion in the project narrative.

- 8.4.3.2 Low level soil sample - If a smaller volume preserved soil aliquot is provided by the client, a dilution analysis may be performed. Depending on the dilution factor, sample weight down to 0.5 gram or a 10X dilution may be used. Any dilution more than 10X, using less than 0.5 gram will necessitate using the medium level methanol preserved approach below.
- 8.4.3.3 Medium level approach - Depending on the dilution factor, reduce the methanol extract from the ratio of 100 μ L/5mL to as low as 5 μ L/5mL pure water, or from 800 uL to 40 uL per 40 mL vial. Further dilution will require secondary dilution of the 10mL methanol extract. The amount of methanol used per 40mL vial must be noted on the log book along with the final dilution factor to allow for double-checking of dilution calculations. Any secondary dilution used must be clearly described in the logbook. If more space is necessary, the back of the logbook page is to be used, with a note on the front of the page to refer to the back of the page.
- 8.4.3.4 Criteria for reporting dilutions. The final dilution analysis is always reportable. This analysis should have the concentration of the highest compound near or above the mid range (100 ppb level) of the initial calibration.
- 8.4.3.5 If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis is generally also reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the letters "DL" appended to the sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and, if reported on CLP-type forms, qualified with a "D" if detected and reported in the more dilute (DL) analysis.
- 8.4.3.6 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the

calibration range of the instrument the initial analysis may be performed at dilution. This information may include project history, prior analyses, screening results, results of other (such as GRO or TPH) analyses, solvent or petroleum odors detected during other analyses or during sampling, etc. This information should be used to prevent overloading and contamination of the autosampler/purge and trap system. If the initial analysis is performed at dilution, and the results of this analysis are acceptable (at or above the mid range calibration standard, or significant non-target compound concentrations), a less dilute analysis is not required. The sample ID is not changed by adding "DL", but the initial analysis at dilution is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.4.3.7 If the initial analysis fails QC criteria, it is not typically reported. If only the dilution analysis is reported, the letters DL are not added to the sample ID, but the dilution is noted on the data review checklist submitted with the data for review. The letters "DL" indicate a second dilution, not an initial analysis at dilution.

8.4.3.8 If the initial and dilution analysis together demonstrate matrix interference, such as with surrogate/internal standard recoveries out of limit in both analyses, both runs are typically reported. Also if the initial analysis provides important information to the project, it should be reported, with the QC exceedences noted on the data sheets (flagged surrogates on Form 2, flagged internal standards on Form 8, "E" qualifiers on Form 1, etc) and fully described in the data review checklist included with the data submitted for review so they may be included in the project narrative.

8.4.4 Acceptance Criteria for Sample Analysis are as follows:

- The sample must meet analysis holding time.
- The sample has to have a compliant tune, initial calibration and continuing calibration.
- The sample has to have a compliant method blank.
- The sample has to have a compliant LCS.
- All surrogate recoveries are within control limits (See **section 10.4**) with the exception of one outlier, unless specified in client project. The outlier must have recovery value above 10%.
- The internal standard areas must meet the -50% to +100% criteria. If the criteria are not met, the sample should be rerun. In some circumstances (high TIC content) this is not necessary, see the corrective action table, **Attachment 8** of this SOP.

- The relative retention time (RRT) of each of the IS must not shift more than ± 0.06 RRT units from the CCV or the mid-point standard of initial calibration.
- All of the target analyte concentration should be below the calibration range excluding the “solvent” front, no ion should saturate the detector
- If the previous run contains any target analyte above the calibration range, and the same target analyte is detected above the reporting limit in the subsequent run, the subsequent run has to be repeated to demonstrate that the compound is not due to carry-over.

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative retention time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$\text{RRT} = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Comparability of mass spectra - The requirements for qualitative verification by comparison of mass spectra is as follows:

- All ions present in the standard mass spectra at a relative abundance greater than 25% must be present in the sample spectrum.
- The relative intensities of ions specified above must agree within $\pm 20\%$ [method allows 30%] between the standard and sample spectra.
- Ions greater than 10.0% in the sample spectrum but not present in the standard spectrum must be considered; this may be due to potential co-eluting interference.
- The halogenated target analytes should contain the characteristic chlorine and bromine isotopic ratios.
- If the criteria above are not met but in the technical judgment of the analyst that the identification is correct, the lab will report the identification and proceed with the quantitation. Any suspect identification should be described on the data review checklist submitted with the data for review.

9.2 Identification of non-target compounds [tentatively identified compounds (TICs)] - Client may request the analysis of TICs. Non-target compounds will be searched using the NIST/EPA/NIH library. The non-target compound will be reported as part of the analysis requirement if:

9.2.1 The client requires a full data package deliverable, including CLP, Mitkem Level 4 or New York ASP-B reporting format (exceptions are projects that have a short list of target analytes such as TCLP, BTEX, STAR list or projects that the client specified no TIC reporting).

The non-target compounds will be identified and reported if:

- Its response is greater than 10% of the closest eluting interference free internal standard.
- Its retention time is within the range of 30 seconds before the elution of the first target compounds, and 3 minutes after the elution of the last target compound.
- Unless specified, up to **10** TIC are to be reported.

9.2.2 Guidelines for making tentative identification :

- Ions greater than 10% in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in sample spectrum but not in the reference spectrum should be reviewed for co-eluting interferences.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed with caution because of background contamination and/or co-eluting interferences.
- The lab shall not report semivolatile target compounds.
- The non-target compounds will be reported as “unknown” if no valid tentative identification can be made (as based on analysts’ interpretation).
- If the Quality (Qual) of the match as determined by the library search program is above 85%, it typically meets the criteria above, and is considered a tentative identification. If the Qual is less than 85%, the match typically does not meet the criteria above, and is usually identified as “unknown”.

9.3 Quantitation of target compounds - The initial calibration is used to quantitate the target compounds. It is important to note that the concentrations of the target compounds not exceed the calibration range of 200 $\mu\text{g/L}$ ($\mu\text{g/kg}$) for all compounds other than the m- and p-xylenes (at 400 $\mu\text{g/L}$ or $\mu\text{g/kg}$) in the analyses of 5 mL water or 5 g soil samples. In the case of 25 mL water sample analyses, the concentrations for all the compounds should be less than or equal to 40 $\mu\text{g/L}$. Any target analyte concentration that exceeds the calibration range will be diluted and reanalyzed.

9.3.1 Manual integration will be performed if needed and documented according to the current revision of SOP 110.0008, Manual Integration of IC, GC and

GC/MS Chromatographs. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. Hardcopies of the ion chromatogram of the primary and secondary ion will be generated. The analyst will further initial and date the manual integration with the proper reason code per SOP 110.0008, Manual Integration of IC, GC and GC/MS Chromatographs.

9.3.2 Determining the concentration of Target Compounds - Target compounds identified are quantitated using the following equation:

9.3.1.1 Aqueous concentrations are calculated using the equation:

$$Conc = \frac{(Ax)(Is)(V_0)}{(Ais)(RRF)(V_s)}$$

where: *Conc* = sample concentration in µg/L

Ax = area of the characteristic ion for the compound to be measured

Ais = area of the characteristic ion of the associated internal standard

Is = concentration of internal standard in µg/L

V₀ = purge volume, 5 for 5 mL water sample and 25 for 25 mL water sample

V_s = sample volume analyzed in mL

RRF = relative response factor

9.3.1.2 Soil concentrations are calculated using the equation below:

$$Conc. = \frac{(Ax)(Is)(5)(1000)}{(Ais)(RRF)(S)(W)}$$

Medium Level:

$$Conc = \frac{(Ax)(Is)(V_i)(5)(Df)(1000)}{(Ais)(RRF)(S)(W)(V_a)}$$

where: *Conc* = Sample concentration in µg/Kg.

Ax = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion of the associated internal standard
 I_s = concentration of internal standard in $\mu\text{g/L}$
 D_f = dilution factor, typically is equal to 1; if there is a secondary dilution, the dilution factor refer to the dilution between the first and the secondary dilution
 RRF = relative response factor
 V_t = total volume of methanol extract, in mL
 V_a = volume of the aliquot of the sample methanol extract, in mL
 S = solid content expressed in decimal value
 W = sample weight added to purge tube or for extraction, in gram

Solid sample results will be reported at dry weight basis unless otherwise specified. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids (see [SOP 110.0038 Percent Solids Determination](#))

9.4 Determining the concentration of non-target compounds - An estimated concentration for non-target compounds is determined using the closest eluting internal standard. The formula to calculate the concentration is the same as those for water and soil samples described above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the associated internal standard. A RRF of one (1) is assumed. An estimated concentration must be calculated for all tentatively identified compounds as well as these identified as unknown.

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery (\%R)} = 100 \times (\text{SSR} - \text{SR}) / (\text{SA})$$

where: SSR = spiked sample result
SR = sample concentration

SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1 - D2)}{(D1 + D2)/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

- 10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. All analysts must have read this SOP and asked questions and received explanation for any areas they are unsure of. This SOP should be referred to often, and used as a reference for this procedure. Details of the procedure for documenting analyst proficiency can be found in the current revision of SOP 80.0016.
- 10.2 Method Blanks - Method Blanks are analyzed to determine the level of contamination associated with the processing and analysis of samples.

10.2.1 Frequency of Method Blank

- The Method Blank must be analyzed after each initial calibration and during each 12-hour time period when the instrument is used for analysis.
- The Method Blank must be analyzed after the Continuing Calibration and before any samples are analyzed.

10.2.2 Procedure for Method Blank:

The Method Blank is analyzed using 5 mL of organic-free water that is spiked with 1 μ L combined IS/SS (Internal Standard/Surrogate Standard) to give a final concentration of 50 μ g/mL. For 25mL purge analysis, the sample is spiked with 5 μ L of the IS/SS solution to yield a final concentration of 5 μ g/L. Blanks are analyzed as ambient purge for aqueous/medium soil samples. For low soil analysis, 5.0g of VOA-free Ottawa sand will be weighed into a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water and analyze by the heated purge procedure. The auto-sampler adds the IS/SS solution automatically.

10.2.3 Acceptance criteria for Method Blank:

- Percent recovery of surrogate must be within the control limits listed in **Section 10.4**.
- All internal standard response must be within the -50% to +100% criteria. If the criteria are not met, the blank should be rerun.
- The concentration of each target compound found in the Method Blank must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10 µg/L or 10 µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1 µg/L, and acetone and 2-butanone must be less than 10 µg/L).

For *DoD* projects, the concentration of the target compounds in the method blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

Any Method Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze the blank. There may be situations where other corrective actions are appropriate depending on project-specific criteria, such as when the sample analysis resulted in a non-detect for the compound that failed the blank acceptance criteria.

10.2.4 All compounds present in method blanks that are also present in samples will be qualified with a “B” flag on data sheets reported to the client. The meaning of this qualifier will be described in the report. This will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

10.3 Storage Blanks- Storage Blanks are analyzed to determine the level of contamination associated with the storage of samples. They are analyzed as a sample at the end of the analytical sequence.

10.3.1 Acceptance criteria for Storage Blanks:

- The concentration of each target compound found in the Storage Blanks must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10 µg/L or 10 µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1 µg/L, and acetone and 2-butanone must be less than 10 µg/L).

For *DoD* projects, the concentration of the target compounds in the Storage Blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

- Any Storage Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze another Storage Blank. If the reanalysis confirms the contamination, the situation must be investigated and the affected client(s) must be notified of the potential contamination issue in the applicable refrigerator.

10.4 Laboratory Control Sample (LCS) -One LCS is prepared with each batch of up to 20 samples of the same matrix. The LCS is spiked with all compounds being reported for the method. If a non-routine compound is being reported, but no LCS is available, this must be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

- For an aqueous LCS sample, mixed standards are spiked into a 40 mL vial of organic-free water, resulting in concentrations at the mid-level standard. See **Table 2** for details on spiking volumes and solutions.
- For a solid LCS sample, add 5.0g of VOA-free Ottawa sand to a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water to the vial. Then add the commercially prepared standards with known values of VOC concentrations by spiking standards into the vial and

analyzing by the heated purge procedure. Where applicable, a Lab Control Sample Duplicate (LCS D) will also be performed to evaluate reproducibility.

10.4.1 Acceptance criteria for LCS:

- Percent recovery of surrogates must be within the control limits listed in **Section 10.4**. For regular SW8260 projects, the recovery is evaluated against the established in-house limits. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits. See **Attachment 4** for *DoD* QC limits.
- All internal standard response must be within the -50% to +100% criteria. If the criteria are not met, the LCS should be rerun.
- If target analytes are outside of the acceptance limits, corrective action is required. Project-specific requirements, if available, will dictate the corrective action performed. See **Attachment 8** for further guidance.
- Due to the large number of target analytes, some recoveries (up to 5 for full list) may be out. These outliers are to be sporadic failures. Compounds that constantly fail to meet criteria require corrective action and investigation.

Per *DoD* requirements, analyses of <11 analytes, no marginal exceedences (ME) are allowed. For the analysis of 11-30 analytes, one ME is allowed; for the analysis of 31-50 analytes, two ME are allowed; for 51-70 analytes, (typical 8260 analysis) three ME are allowed; for the analysis of 71-90 analytes, four ME are allowed; for the analysis of >90 analytes, five ME are allowed. See **Attachments 5 and 6** for further guidance.

- Reporting LCS Results – If any compounds are outside of the acceptance limits, their recoveries are qualified with the “*” flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged with an “S” on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries - The recovery of the surrogate compounds (also called System Monitoring Compounds) in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.1 Acceptance criteria - The percent recovery of each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS must be within the in-house acceptance window with the exception of one surrogate per fraction

allowed out. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits.

- For *DoD* projects, values from QSM:

	<u>Solid</u>	<u>Aqueous</u>
• 1,2-dichloroethane-d4	no limits given	70-120
• dibromofluoromethane	no limits given	85-115
• toluene-d8	85-115	85-120
• 4-bromofluorobenzene	85-120	75-120

10.5.2 Corrective action - If the recovery of the system monitoring compound is out of the acceptable window in the method blank or LCS, corrective action must be implemented. Corrective action should include verification of the Internal standard area integrations, checking for errors in calculations and confirming the use of appropriate standards. In addition, the blank and/or LCS may be re-analyzed. If the recovery of the system-monitoring compound is outside of the acceptance limit for a sample, the data will be evaluated and corrective action (commonly reanalysis) will be taken. See **Attachment 8** for corrective action guidelines.

10.5.3 Reporting – The Target data reduction and reporting programs will flag any surrogate recovery outside of the acceptance limits with a “*”; the LIMS Level 2 reporting will flag any surrogate recovery outside of the acceptance limits with a “S”. If the sample is reanalyzed and the system monitoring compounds are within the acceptance criteria for the reanalysis, and the reanalysis is within holding time, report the results of the reanalysis only. If the same system monitoring compounds are out in the reanalysis, report both sets of analysis results to demonstrate matrix-related problems. This should be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

10.6 Matrix spike/matrix spike duplicate samples (MS/MSD) are analyzed at a frequency of once per twenty samples of similar matrix and procedure. The duplicate matrix spikes are used to assess the effect of matrix on the analytical accuracy and precision for the batch of samples. Where the client has not provided sufficient sample aliquots for a MS/MSD to be included in every batch, a duplicate LCS should be performed so analytical precision can be demonstrated. The duplicate matrix spikes are typically spiked with all of the target analytes. There may be project-specific MS spiking lists and criteria which take precedence.

- 10.6.1 The percent recovery of each compound is compared to the in-house acceptance limits, project-specific limits or **Attachment 6**. These limits are the same as used for LCS, but are used as advisory guidelines.
- 10.6.2 The following factors could greatly affect the accuracy and precision of the matrix spikes and matrix spike duplicates: sample heterogeneity, much higher analyte concentration in the sample, and matrix effect. The best measurement is obtained if the spike concentration is two to four times the analyte concentration in the unspiked sample.
- 10.6.3 If target compound recoveries are outside of the MS acceptance limits corrective action is required. See **Attachment 8** of this SOP for corrective action guidelines. Evaluate the percent recovery for those compounds outside of the recovery limit to the same compound in the LCS. At a minimum the corrective action will involve flagging any MS value outside of the control limit with an "*" on the recovery summary report form (Form 3) or an "S" on the LIMS Level 2 report. This is also noted on the data review checklist submitted with the data for review to allow for inclusion in the report narrative. Other corrective actions may include reanalysis of the MS/MSD at a higher spiking concentration, reanalysis of the MS/MSD by dilution of the sample, discussion of the issue with the Project Manager and the client.

For *DoD* projects, the %RPD limits for the duplicate set is 30%.

- 10.7. MDL studies are conducted to establish the detection limits applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD QSM Version 4.1 Limit of Detection (LOD). MDL verification must be analyzed quarterly on each instrument used for DoD program work. Please refer to the Mitkem SOP No. 80.0005 Determination of Method Detection Limits for more detail.

11. Data Validation and Reporting

All raw data, including calibrations, QC results, and samples results, are reviewed for technical accuracy and completeness. The guidelines and procedures taken to ensure the data quality is listed in Section 11 of Quality Assurance Plan.

12. Corrective Action Procedures

- 12.1 All QC exceedences require a corrective action response and documentation. The proper corrective action depends on the specific situation. Many actions are spelled-out in this SOP. A table describing common occurrences, corrective actions and documentation is attached as **Attachment 8**.

12.2 Further information on Mitkem's corrective action policy and procedures are included in the current revision of SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. MSDS sheets were kept in safety officer's recover. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and safety glasses when handling these reagents and work in a hood whenever possible.

13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8260C, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 3 August 2006.
2. Mitkem Laboratories Quality Assurance Plan (QAP), current revision.
3. "Quality Systems Manual for Environmental Laboratories" Department of Defense, Final Version 4.1 April 2009.
4. Environmental Protection Agency. Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples Method 5035, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 0, December 1996.
5. Environmental Protection Agency. Purge-and-Trap for Aqueous Samples Method 5030, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 2, December 1996.
6. Hewlett Packard and OI Analytical Instrument instruction manuals.
7. ASTM D6418 - 04 Standard Practice for Using the Disposable EnCore Sampler for Sampling and Storing Soil for Volatile Organic Analysis.

16. Low Level Calibration and Analysis

1. A low level calibration procedure is used to achieve a low level reporting limit at 0.5 ug/L for most of the target analytes, except for ketones. The purge volume for all standards, QC samples and field samples are 25 mL instead of 5 mL. The 5 level calibration standards are 0.5, 4.0, 10, 20 and 40 ug/L, except for ketone compounds at 5.0, 40, 100, 200 and 400ug/L.
2. Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 200 uL each of the IS stock solution (Cat. No. 30241) and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3600 uL of methanol to make a 125 ug/mL solution. This solution once prepared is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 25 mL purge volume, this yields a concentration of 5 ug/L.
3. The working standard for the calibration standards is prepared by diluting the 5 mL analysis calibration standard 5 times to a concentration of 20 ug/mL. Additional ketone standards are added at this step to result in a concentration of 100 ug/mL for the ketone compounds.
4. The 5 level initial calibration standards are prepared by adding 1, 8, 20, 40 and 80 uL of the calibration standard, 1, 8, 20, 40 and 80 uL of the surrogate standard and 10 uL of the internal standard to each 40 mL DI water. 25 mL of each solution are used for calibration analysis. Initial calibration criteria are the same as the 5 mL analysis.
5. A second source Initial Calibration Verification (ICV) is performed after the completion of the multi-level calibration, at 10ug/L. The calculated value of the analytes in the ICV should be 70 – 130% of the expected value (7.0-13.0 ng/uL). DoD limits are 80-120%.
6. The continuing calibration standard is prepared by adding 20 uL of the calibration standard, 20 uL of the surrogate standard and 10 uL of the internal standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency and criteria of continuing calibration are the same as the 5 mL analysis.
7. Method blank is prepared by adding 1uL of the IS/SS standard to a 25 mL DI water. The frequency and criteria of method blank are the same as 5 mL analysis. No target compound can be detected above one half of the required reporting limits, except for Methylene Chloride which must be less than 2 ug/L.
8. LCS is prepared by adding 20 uL of the calibration standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency of LCS is the same as 5 mL analysis. Recovery criteria are based on in-house limits and can be found in LIMS.

9. Samples are analyzed after all calibration and QC samples have been analyzed and passed their criteria. Each sample is spiked with 1uL of the IS/SS standard by the autosampler. 25 mL of each spiked sample is used for analysis. The criteria for sample analysis are the same as the 5 mL analysis.
10. MS/MSD samples are spiked with 20 uL of the calibration standard. 25 mL of each spiked sample is used for analysis and is spiked with 1uL of the IS/SS standard by the autosampler. The frequency and criteria of MS/MSD samples are the same as the 5 mL analysis. However, since the whole sample vial is spiked and used for each analysis, MS/MSD for 25 mL analysis can only be performed when there are three or more sample vials available for the designated sample.

Attachments:

Table 1: List of Abbreviations

Table 2: Working Standard / LCS Detail.

Table 3: Suggested minimum RFs (Table 4 from Published Method).

Figure 1: LIMS standard/spike Logbook, Main page .

Figure 2: LIMS standard/spike Logbook, Analyte page.

Figure 3: Instrument Run Logbook.

Attachment 1: SW8260 Target Analyte List

Attachment 2: BFB Tune Chromatogram and Mass Listing.

Attachment 3: BFB Tune Mass Spectrum and Ion Abundance Criteria.

Attachment 4: Chromatograph and Quantitation Report of 50ug/L Standard.

Attachment 5: DoD Specific QC Requirements: Table F-4.

Attachment 6: DoD Specific QC Control Limits, Tables G-4 and G-5.

Attachment 7: Additional QA/QC Requirements for MA_DEP

Attachment 8: Corrective Action and Documentation Examples.

Table 1
List of Abbreviations

BFB	Bromofluorobenzene
DoD	Department of Defense (includes Army, Navy, Air Force)
LCS	Lab control sample
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method detection limit
MQL	Method quantitation limit
ME	Marginal Exceedence
MS	Matrix spike
MSD	Matrix spike duplicate
QSM	Quality Systems Manual for DoD work
RL	Reporting Limit (occasionally referred to as PQL or Practical Quantitation Limit {in the LIMS}, or MRL or Method Reporting Limit)

Table 2
Working Standard / LCS Detail
Method SW8260

Aqueous											
uL spike amounts for 40mL Vials											
	QC				Sample	ICAL (Concentration)					
	CCV	Blank	LCS	MS/MSD		5	20	50	100	200	1
GAS	20	-	20	20	-	2	8	20	40	80	0.4
STD	20	-	20	20	-	2	8	20	40	80	0.4
APPIX	20	-	20	20	-	2	8	20	40	80	0.4
SS*	20	20	20	20	20	2	8	20	40	80	0.4
IS*	20	20	20	20	20	20	20	20	20	20	20

Soil										
uL spike amounts for 5mL H ₂ O										
	QC				Sample	ICAL (Concentration)				
	CCV	Blank	LCS	MS/MSD		5	20	50	100	200
GAS	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
STD	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
APPIX	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
SS*	2.5	2.5	2.5	2.5	2.5	0.25	1	2.5	5	10
IS*	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

Blank and LCS vials should contain approx. 5g VOC-free soil preserved in 5mL D.I. Water

*These may be machined spiked for samples, CCV and some project ICALS

Table 3
Suggested minimum RFs
(Table 4 from Published Method)

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Volatile Compounds	Minimum Response Factor (RF) ^a	Typical Response Factor (RF) ^b
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

^a The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

^b Data provided by EPA Region III laboratory.

Figure 1

LIMS standard/spike Logbook
 Main page

Solution ID: VW100225C **Soln Type:** Standard **Type:** Intermediate

Solution Name: 8260 ICV **Status:** New **Solvent Lot:** CY840

Prepared BY: Ashley Marquis **Dept:** IMSVCA **Solvent:** Methanol

Date Prepared: 2/25/2010 **Expiration Date:** 3/25/2010

Final Volume (mL): 4

Stock ID	Stock Name	Amt Used	Units
VP090317D	8260 ICV CASES H-502B-10X	200.000	µL
VP090806B	8260 CUSTOM ICV STANDARD	1000.000	µL
VP090317B	8260 ICV KETONES CLP-022K-10X	200.000	µL
VP090806B	8260 STD ICV H-502A-R-10X	200.000	µL

Record: 14 of 4

Figure 2

LIMS standard/spike Logbook
 Analyte page

The screenshot displays the 'Standards' window in the Milkem LIMS software. The window title is 'Milkem LIMS - [Standards]'. The menu bar includes 'File', 'Edit', 'Insert', 'Records', 'Window', and 'Help'. The toolbar contains various icons for file operations and data management. The main interface is divided into several sections:

- Left Panel:** A vertical list of standard identifiers (e.g., VU100225C, VU100217A, VU100211A) categorized by 'Dept', 'MSVDA', 'Saln', 'Standards', 'Spikes', 'Both', 'TYPE', 'Primary', 'Intermedia', 'Working', 'Heat', 'Other', 'All', 'Status', 'Current', 'Past', 'Print', 'Label', 'Copy', and 'Find Std'.
- Top Section:** Fields for 'Solution ID' (VU100225C), 'Soln Type' (Standard), and 'Type' (Intermediate). Below these are 'Instrument Spike' and 'Units' (ug/mL).
- Main Table:** A table titled 'Main Analytes' with columns: AT, Analyte, CAS, Final Conc, VendorID, and LotNumber. The table lists 20 different chemical analytes, each with its corresponding CAS number, concentration, vendor, and lot number.
- Bottom Section:** A 'Record' indicator showing '1 of 93'.

AT	Analyte	CAS	Final Conc	VendorID	LotNumber
T	Chloroethane	75-00-3	100.000	AccuStandard	B8080260
T	Bromomethane	74-83-9	100.000	AccuStandard	B8080260
T	Chloromethane	74-87-3	100.000	AccuStandard	B8080260
T	Dichlorodifluoromethane	75-71-8	100.000	AccuStandard	B8080260
T	Trichlorofluoromethane	75-69-4	100.000	AccuStandard	B8080260
T	Vinyl Chloride	75-01-4	100.000	AccuStandard	B8080260
A	1,1,2-Trichloro-1,2,2-trifluoroethyl	76-13-1	100.000	ACCUSTAND	B8020093-1A
A	1,4-Dioxane	123-91-1	200.000	ACCUSTAND	B8020093-1A
A	1-Chlorohexane	544-10-5	100.000	ACCUSTAND	B8020093-1A
A	Acetonitrile	75-05-8	100.000	ACCUSTAND	B8020093-1A
A	Acrolein	107-02-8	100.000	ACCUSTAND	B8020093-1A
A	Acrylonitrile	107-13-1	100.000	ACCUSTAND	B8020093-1A
A	Allyl chloride	107-05-1	100.000	ACCUSTAND	B8020093-1A
A	Carbon disulfide	75-15-0	100.000	ACCUSTAND	B8020093-1A
A	Cyclohexane	110-82-7	100.000	ACCUSTAND	B8020093-1A
A	Diethyl ether	60-29-7	100.000	ACCUSTAND	B8020093-1A
A	Diisopropyl ether	108-20-3	100.000	ACCUSTAND	B8020093-1A
A	Ethanol	64-17-5	10000.000	ACCUSTAND	B8020093-1A
A	Ethyl methacrylate	97-63-2	100.000	ACCUSTAND	B8020093-1A
A	Ethyl tert-butyl ether	637-92-2	100.000	ACCUSTAND	B8020093-1A
A	Hexachloroethane	67-72-1	100.000	ACCUSTAND	B8020093-1A
A	Iodomethane	74-89-4	100.000	ACCUSTAND	B8020093-1A
A	Isobutyl alcohol	78-83-1	200.000	ACCUSTAND	B8020093-1A
A	Methacrylonitrile	126-98-7	100.000	ACCUSTAND	B8020093-1A
A	Methyl acetate	79-20-5	100.000	ACCUSTAND	B8020093-1A
A	Methyl methacrylate	80-62-6	100.000	ACCUSTAND	B8020093-1A

Figure 3

Instrument Run Logbook

Attachment 1 Target Analyte List for 8260

Method 8260 Target Analyte List

type	Target Analyte	type	Target Analyte
A	1,1,1,2-Tetrachloroethane	I	1,4-Dichlorobenzene-d4
A	1,1,1-Trichloroethane	I	Chlorobenzene-d5
A	1,1,2,2-Tetrachloroethane	I	Fluorobenzene
A	1,1,2-Trichloroethane	S	1,2-Dichloroethane-d4
A	1,1-Dichloroethane	S	Bromofluorobenzene
A	1,1-Dichloroethene	S	Dibromofluoromethane
A	1,1-Dichloropropene	S	Toluene-d8
A	1,2,3-Trichlorobenzene		
A	1,2,3-Trichloropropane	X	1,1,2,2-Tetrachloroethane-d2
A	1,2,4-Trichlorobenzene	X	1,1,2-Trichloro-1,2,2-trifluoroethane
A	1,2,4-Trimethylbenzene	X	1,1-Dichloroethene-d2
A	1,2-Dibromo-3-chloropropane	X	1,2-Dichlorobenzene-d4
A	1,2-Dibromoethane	X	1,2-Dichloroethene, Total
A	1,2-Dichlorobenzene	X	1,2-Dichloropropane-d6
A	1,2-Dichloroethane	X	1,2-Dichlorotetrafluoroethane
A	1,2-Dichloropropane	X	1,3,5-Trichlorobenzene
A	1,3,5-Trimethylbenzene	X	1,4-Difluorobenzene
A	1,3-Dichlorobenzene	X	1,4-Dioxane
A	1,3-Dichloropropane	X	1-Chlorohexane
A	1,4-Dichlorobenzene	X	2-Butanone-d5
A	2,2-Dichloropropane	X	2-Chloro-1,3-butadiene
A	2-Butanone	X	2-Chloroethyl vinyl ether
A	2-Chlorotoluene	X	2-Ethyl-1-hexanol
A	2-Hexanone	X	2-Hexanone-d5
A	4-Chlorotoluene	X	2-methyl-2-propanol
A	4-Isopropyltoluene	X	Acetonitrile
A	4-Methyl-2-pentanone	X	Acrolein
A	Acetone	X	Acrylonitrile
A	Benzene	X	Alkylbenzenes, Total
A	Bromobenzene	X	Allyl chloride
A	Bromochloromethane	X	Benzene-d6
A	Bromodichloromethane	X	bis(2-Chloroethyl)ether
A	Bromoform	X	Bromoform-d
A	Bromomethane	X	Chloroethane-d5
A	Carbon disulfide	X	Chloroform-d
A	Carbon tetrachloride	X	Cyclohexane
A	Chlorobenzene	X	Diethyl ether
A	Chloroethane	X	Dilsopropyl ether
A	Chloroform	X	Ethanol
A	Chloromethane	X	Ethyl methacrylate
A	cis-1,2-Dichloroethene	X	Ethyl tert-butyl ether
A	cis-1,3-Dichloropropane	X	Freon-113
A	Dibromochloromethane	X	Hexachloroethane
A	Dibromomethane	X	Isobutyl alcohol
A	Dichlorodifluoromethane	X	Isopropyl alcohol
A	Ethylbenzene	X	Methacrylonitrile

Attachment 1(cont'd)
Target Analyte List for 8260

A	Hexachlorobutadiene	X	Methyl acetate
A	Iodomethane	X	Methyl methacrylate
A	Isopropylbenzene	X	Methylcyclohexane
A	m,p-Xylene	X	Pentachloroethane
A	Methyl tert-butyl ether	X	Pentafluorobenzene
A	Methylene chloride	X	Propionitrile
A	n-Butylbenzene	X	tert-Amyl methyl ether
A	n-Propylbenzene	X	Tetrahydrofuran
A	Naphthalene	X	trans-1,3-Dichloropropene-d4
A	o-Xylene	X	trans-1,4-Dichloro-2-butene
A	sec-Butylbenzene	X	Vinyl chloride-d3
A	Styrene	X	Xylenes (Total)
A	tert-Butylbenzene		
A	Tetrachloroethene		
A	Toluene		
A	trans-1,2-Dichloroethene		
A	trans-1,3-Dichloropropene		
A	Trichloroethene		
A	Trichlorofluoromethane		
A	Vinyl acetate		
A	Vinyl chloride		

Type	Explanation
A	Analyte (routine)
I	Internal Standard
S	Surrogate
X	Analyte (non-routine)

Attachment 2 BFB Tune Chromatogram

Data File: \\AVOCADRO\ORGANICS\organic\voa\V6.1\060201A.B\V6E0620.D

Page 1

Date: 01-FEB-2006 12:27

Client ID: BFB6Z

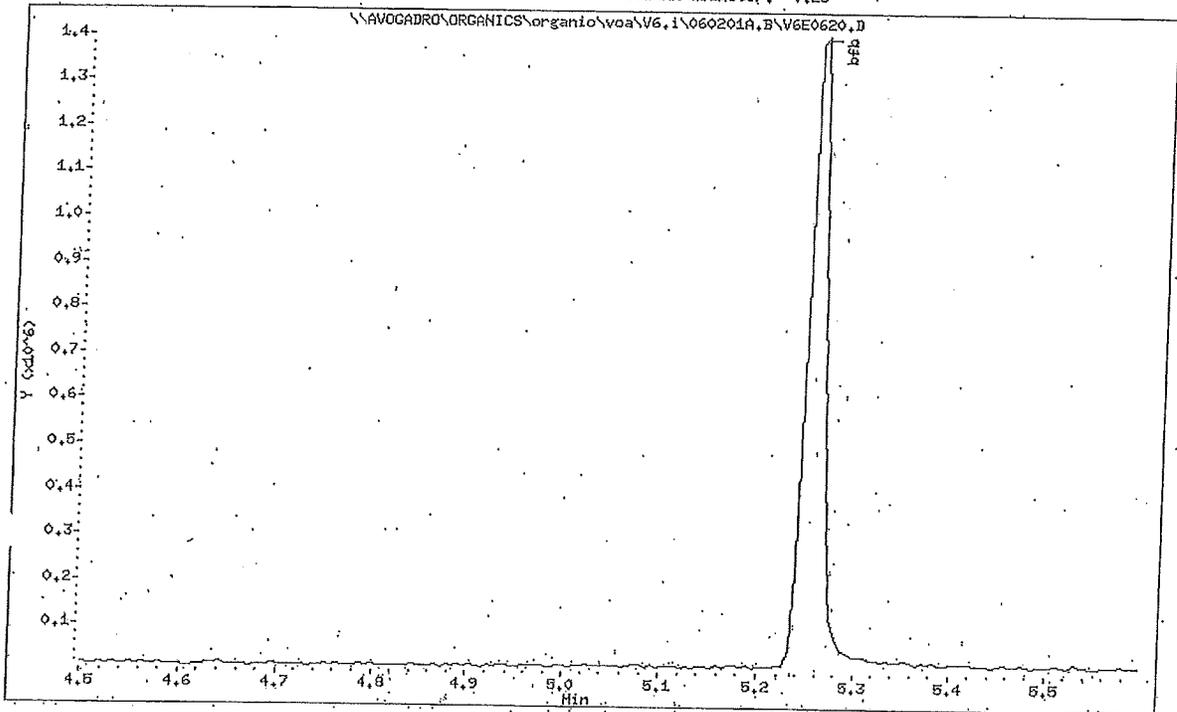
Instrument: v6.1

Sample Info: 2ul,BFB6Z,BFB6Z,

Operator: LG

Column phase: DB-624

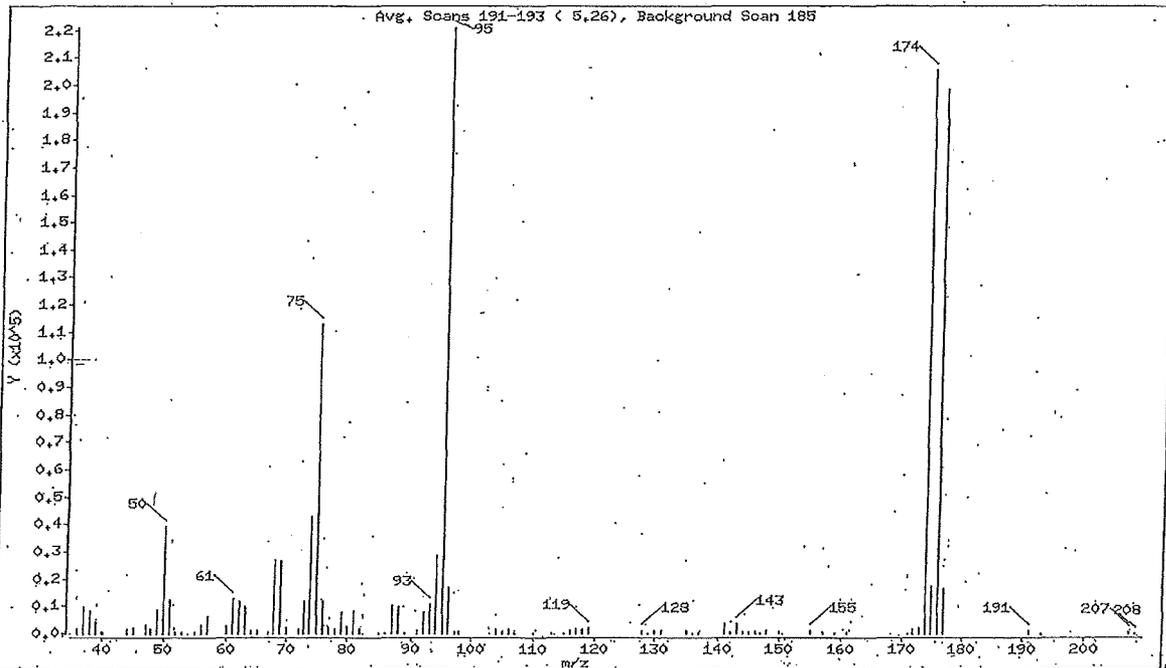
Column diameter: 0.25



Attachment 3

BFB Tune Mass Spectrum and Ion Abundance Criteria

Data File: \\AVOGADRO\ORGANICS\organic\voa\V6.i\060201A,B\V6E0620.D Page 2
 Date: 01-FEB-2006 12:27
 Client ID: BFB6Z Instrument: v6.i
 Sample Info: 2ul,BFB6Z,BFB6Z, Operator: LG
 Column phase: DB-624 Column diameter: 0.25
 i bfb



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
95	Base Peak, 100% relative abundance	100.00
50	15.00 - 40.00% of mass 95	17.82
75	30.00 - 60.00% of mass 95	51.26
96	5.00 - 9.00% of mass 95	7.72
173	Less than 2.00% of mass 174	1.03 (< 1.11)
174	50.00 - 100.00% of mass 95	93.13
175	5.00 - 9.00% of mass 174	7.74 (< 8.36)
176	95.00 - 101.00% of mass 174	89.94 (< 96.82)
177	5.00 - 9.00% of mass 176	7.42 (< 8.25)

Data File: \\AVOGADRO\ORGANICS\organic\voa\V6.1\060201A.B\V6E0620.D

Page 3

Date: 01-FEB-2006 12:27

Client ID: BFB6Z

Instrument: v6.i

Sample Info: 2ul,BFB6Z,BFB6Z,

Operator: LG

Column phase: DB-624

Column diameter: 0.25

Data File: V6E0620.D
 Spectrum: Avg. Scans 191-193 (5,26), Background Scan 185
 Location of Maximum: 95.00
 Number of points: 94

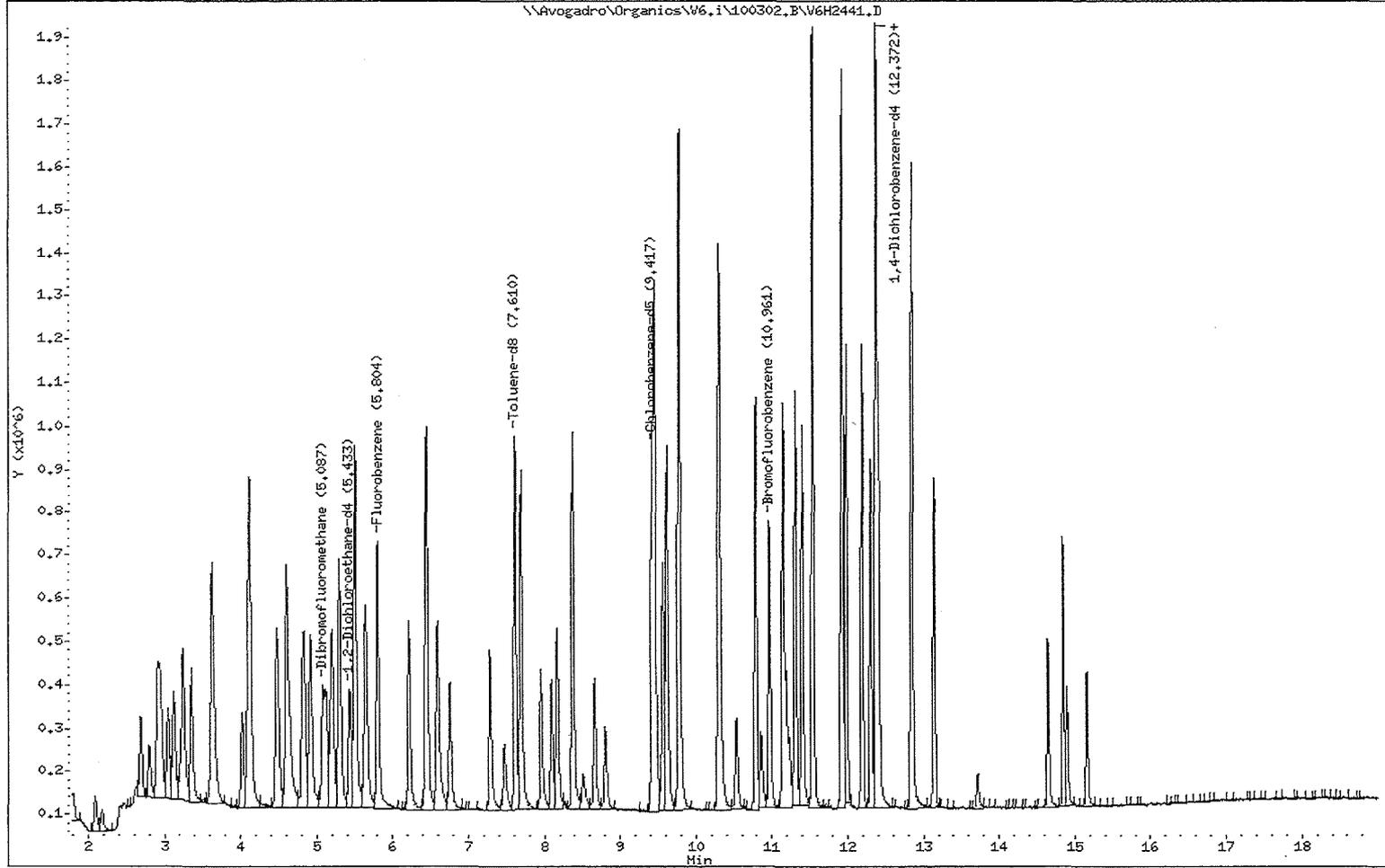
m/z	Y	m/z	Y	m/z	Y	m/z	Y
36.00	1702	68.00	26928	96.00	17040	143.00	3728
37.00	9822	69.00	26472	97.00	644	144.00	398
38.00	7957	70.00	1977	98.00	258	145.00	624
39.00	4095	72.00	1394	103.00	202	146.00	379
40.00	472	73.00	11595	104.00	1583	147.00	189
44.00	1567	74.00	42872	105.00	398	148.00	812
45.00	2280	75.00	113192	106.00	1441	150.00	287
47.00	2965	76.00	11922	107.00	172	155.00	936
48.00	1681	77.00	1906	110.00	236	157.00	479
49.00	8314	78.00	1301	113.00	169	159.00	119
50.00	39344	79.00	7593	115.00	227	161.00	288
51.00	12361	80.00	2364	116.00	1139	171.00	174
52.00	506	81.00	8306	117.00	1693	172.00	1756
53.00	289	82.00	1472	118.00	1309	173.00	2279
55.00	538	85.00	167	119.00	2109	174.00	205760
56.00	3101	86.00	243	128.00	1110	175.00	17098
57.00	6291	87.00	10388	129.00	191	176.00	198592
60.00	2730	88.00	9715	130.00	1050	177.00	16375
61.00	12549	89.00	186	131.00	824	191.00	1084
62.00	11630	91.00	1240	135.00	961	193.00	212
63.00	9920	92.00	7869	136.00	170	207.00	648
64.00	1146	93.00	10870	137.00	701	208.00	107
65.00	1241	94.00	28568	141.00	3681		
67.00	497	95.00	220800	142.00	445		

Attachment 4

Chromatogram and Quantitation Report of 50 µg/L Standard

Data File: \\Avogadro\Organics\V6.i\100302.B\V6H2441.D
Date : 02-MAR-2010 10:07
Client ID: VSTD0506Y
Sample Info: 5ML,VSTD0506Y,VSTD0506Y
Purge Volume: 5.0
Column phase: DB-624

Instrument: V6.i
Operator: SZ SRC: SZ
Column diameter: 0.25



Data File: \\Avogadro\Organics\V6.i\100302.B\V6H2441.D
 Report Date: 19-Mar-2010 08:37

Mitkem Laboratories

Method 8260 Water and Medium Soil
 Data file : \\Avogadro\Organics\V6.i\100302.B\V6H2441.D
 Lab Smp Id: VSTD0506Y Client Smp ID: VSTD0506Y
 Inj Date : 02-MAR-2010 10:07
 Operator : SZ SRC: SZ Inst ID: V6.i
 Smp Info : 5ML,VSTD0506Y,VSTD0506Y
 Misc Info :
 Comment :
 Method : \\Avogadro\Organics\V6.i\100302.B\v68260GH.m
 Meth Date : 02-Mar-2010 10:48 sz Quant Type: ISTD
 Cal Date : 23-FEB-2010 19:59 Cal File: V6H2321.D
 Als bottle: 3 Continuing Calibration Sample
 Dil Factor: 1.00000
 Integrator: HP RTE Compound Sublist: all.sub
 Target Version: 4.14
 Processing Host: TARGET102

Concentration Formula: Amt * DF * Uf * 5/Vo * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	ng unit correction factor
Vo	5.000	Sample Volume purged (mL)
Cpnd Variable		Local Compound Variable

Compounds	QUANT SIG				AMOUNTS		
	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
1 Dichlorodifluoromethane	85	1.571	1.571	(0.271)	86448	50.0000	35
2 Chloromethane	50	1.681	1.681	(0.290)	137975	50.0000	47
3 Vinyl Chloride	62	1.808	1.808	(0.312)	120119	50.0000	48
4 Bromomethane	94	2.094	2.094	(0.361)	85674	50.0000	52
5 Chloroethane	64	2.185	2.185	(0.377)	52871	50.0000	53
6 Trichlorofluoromethane	101	2.477	2.477	(0.427)	206785	50.0000	54
127 Ethanol	46	2.623	2.623	(0.452)	87610	5000.00	5800 (Q)
7 Ether	59	2.684	2.684	(0.463)	153987	50.0000	53
8 Acrolein	56	2.793	2.793	(0.481)	213501	250.000	250
9 1,1-Dichloroethene	96	2.909	2.909	(0.501)	163941	50.0000	52
11 Acetone	58	2.946	2.946	(0.508)	28774	50.0000	50 (Q)
10 1,1,2-Trichloro-1,2,2-trifluo	101	2.939	2.939	(0.507)	200665	50.0000	54
12 Iodomethane	142	3.043	3.043	(0.524)	418452	50.0000	54
13 Carbon Disulfide	76	3.116	3.116	(0.537)	581924	50.0000	51
14 Acetonitrile	40	3.231	3.231	(0.557)	197428	500.000	800 (Q)
15 Methyl Acetate	43	3.262	3.262	(0.562)	150871	50.0000	49
16 Methylene Chloride	84	3.353	3.353	(0.578)	207540	50.0000	50
17 tert-Butanol	59	3.262	3.262	(0.562)	13408	100.000	110
19 trans-1,2-Dichloroethene	96	3.627	3.627	(0.625)	186111	50.0000	55
20 Methyl tert-butyl ether	73	3.639	3.639	(0.627)	535611	50.0000	52
21 1,1-Dichloroethane	63	4.016	4.016	(0.692)	311050	50.0000	53
22 Vinyl acetate	43	4.101	4.101	(0.707)	595531	50.0000	56

Data File: \\Avogadro\Organics\V6.i\100302.B\V6H2441.D
 Report Date: 19-Mar-2010 08:37

Compounds	QUANT SIG		AMOUNTS				
	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
23 Diisopropyl Ether	45	4.113	4.113	(0.709)	665909	50.0000	53
24 Ethyl tert-butyl ether	59	4.472	4.472	(0.771)	593563	50.0000	54
25 cis-1,2-Dichloroethene	96	4.600	4.600	(0.793)	222234	50.0000	56
26 2,2-Dichloropropane	77	4.606	4.606	(0.794)	215739	50.0000	54
28 2-Butanone	72	4.618	4.618	(0.796)	27520	50.0000	53 (Q)
29 Bromochloromethane	128	4.837	4.837	(0.833)	128430	50.0000	56
30 Tetrahydrofuran	72	4.898	4.898	(0.844)	47054	100.000	95
31 Chloroform	83	4.922	4.922	(0.848)	334831	50.0000	53
§ 32 Dibromofluoromethane	113	5.080	5.080	(0.875)	225886	50.0000	55
M 27 1,2-dichloroethene, (Total)	100				408345	100.000	110
33 1,1,1-Trichloroethane	97	5.129	5.129	(0.884)	250692	50.0000	52
34 Cyclohexane	56	5.202	5.202	(0.896)	318704	50.0000	53
35 1,1-Dichloropropene	110	5.293	5.293	(0.912)	96722	50.0000	55
36 Carbon Tetrachloride	117	5.311	5.311	(0.915)	238412	50.0000	53
§ 37 1,2-Dichloroethane-d4	102	5.427	5.427	(0.935)	33184	50.0000	50
38 Benzene	78	5.506	5.506	(0.949)	713545	50.0000	53
39 1,2-Dichloroethane	62	5.506	5.506	(0.949)	231137	50.0000	52
40 tert-Amyl methyl ether	73	5.646	5.646	(0.973)	528567	50.0000	54
* 41 Fluorobenzene	96	5.804	5.804	(1.000)	814763	50.0000	
42 Trichloroethene	130	6.217	6.217	(1.071)	216374	50.0000	44
43 Methylcyclohexane	83	6.442	6.442	(1.110)	346043	50.0000	54
44 1,2-Dichloropropane	63	6.436	6.436	(1.109)	209899	50.0000	55
46 Dibromomethane	93	6.576	6.576	(1.133)	134188	50.0000	54
47 1,4-Dioxane	88	6.613	6.613	(1.139)	47689	1000.00	1000
48 Bromodichloromethane	83	6.758	6.758	(1.164)	257225	50.0000	54
49 cis-1,3-Dichloropropene	75	7.281	7.281	(1.255)	302191	50.0000	55
50 4-Methyl-2-pentanone	43	7.470	7.470	(1.287)	174620	50.0000	49
§ 51 Toluene-d8	98	7.610	7.610	(0.808)	834673	50.0000	47
45 2-Chloroethyl vinyl ether	63	7.142	7.142	(1.231)	3577	50.0000	230 (A)
52 Toluene	91	7.689	7.689	(1.325)	347584	50.0000	54
53 trans-1,3-Dichloropropene	75	7.950	7.950	(1.370)	263401	50.0000	53
54 1,1,2-Trichloroethane	97	8.169	8.169	(1.408)	191698	50.0000	53
55 Tetrachloroethene	164	8.376	8.376	(0.890)	176280	50.0000	28
56 1,3-Dichloropropane	76	8.376	8.376	(0.890)	259417	50.0000	49
57 2-Hexanone	43	8.510	8.510	(0.904)	120839	50.0000	45
58 Dibromochloromethane	129	8.662	8.662	(0.920)	248144	50.0000	50
59 1,2-Dibromoethane	107	8.802	8.802	(0.935)	206020	50.0000	49
* 60 Chlorobenzene-d5	117	9.416	9.416	(1.000)	655757	50.0000	
63 1-Chlorohexane	91	9.446	9.446	(1.003)	234387	50.0000	49
61 Chlorobenzene	112	9.453	9.453	(1.004)	563727	50.0000	49
62 1,1,1,2-Tetrachloroethane	131	9.562	9.562	(1.015)	223113	50.0000	49
64 Ethylbenzene	106	9.611	9.611	(1.021)	287469	50.0000	49
65 m,p-Xylene	106	9.775	9.775	(1.038)	695279	100.000	99
66 o-Xylene	106	10.292	10.292	(1.093)	351479	50.0000	49
67 Styrene	104	10.316	10.316	(1.096)	580177	50.0000	49
68 Bromoform	173	10.529	10.529	(1.118)	170670	50.0000	50
69 Isopropylbenzene	105	10.784	10.784	(1.145)	853613	50.0000	49
126 trans-1,4-Dichloro-2-butene	75	10.851	10.851	(1.152)	65869	50.0000	54
§ 70 Bromofluorobenzene	95	10.961	10.961	(1.164)	311410	50.0000	48
72 Bromobenzene	156	11.143	11.143	(0.900)	265999	50.0000	48
71 1,1,2,2-Tetrachloroethane	83	11.143	11.143	(0.900)	250243	50.0000	68
73 1,2,3-Trichloropropane	75	11.186	11.186	(0.904)	220637	50.0000	46
74 n-Propylbenzene	120	11.307	11.307	(0.914)	250994	50.0000	47
75 2-Chlorotoluene	126	11.392	11.392	(0.920)	250173	50.0000	49

Data File: \\Avogadro\Organics\V6.i\100302.B\V6H2441.D
 Report Date: 19-Mar-2010 08:37

Compounds	QUANT SIG		AMOUNTS				
	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ug/L)	ON-COL (ug/L)
77 4-Chlorotoluene	126	11.532	11.532	(0.932)	238266	50.0000	48
76 1,3,5-Trimethylbenzene	105	11.532	11.532	(0.932)	724133	50.0000	48
78 tert-Butylbenzene	119	11.922	11.922	(0.963)	784469	50.0000	55
79 1,2,4-Trimethylbenzene	105	11.982	11.982	(0.968)	739485	50.0000	47
80 sec-Butylbenzene	105	12.189	12.189	(0.985)	939454	50.0000	46
82 1,3-Dichlorobenzene	146	12.299	12.299	(0.994)	462061	50.0000	47
83 4-Isopropyltoluene	119	12.365	12.365	(0.999)	732621	50.0000	44
* 84 1,4-Dichlorobenzene-d4	152	12.378	12.378	(1.000)	336816	50.0000	
85 1,4-Dichlorobenzene	146	12.408	12.408	(1.002)	474132	50.0000	46
M 81 Xylene (Total)	106				1046758	150.000	150
87 1,2-Dichlorobenzene	146	12.834	12.834	(1.037)	459864	50.0000	48
86 n-Butylbenzene	91	12.846	12.846	(1.038)	623940	50.0000	41
88 1,2-Dibromo-3-chloropropane	75	13.716	13.716	(1.108)	28493	50.0000	43
89 1,2,4-Trichlorobenzene	180	14.640	14.640	(1.183)	158386	50.0000	30
90 Hexachlorobutadiene	225	14.841	14.841	(1.199)	133237	50.0000	40
91 Naphthalene	128	14.895	14.895	(1.203)	297630	50.0000	25
92 1,2,3-Trichlorobenzene	180	15.163	15.163	(1.225)	124744	50.0000	27

QC Flag Legend

- A - Target compound detected but, quantitated amount exceeded maximum amount.
- Q - Qualifier signal failed the ratio test.

Attachment 5

DoD-Specific QC Requirements

QSM Table F-4

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation \leq 20%.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<p>1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>SVOCs ≥ 0.050.</p> <p>2. RSD for RFs for CCCs: VOCs and SVOCs $\leq 30\%$ and one option below:</p> <p>Option 1: RSD for each analyte $\leq 15\%$;</p> <p>Option 2: linear least squares regression $r \geq 0.995$;</p> <p>Option 3: non-linear regression—coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	<p><u>1. Average RF for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>SVOCs ≥ 0.050.</p> <p><u>2. %Difference/Drift for all target compounds and surrogates:</u> VOCs and SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).</p>	<p>DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken.</p> <p>Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.</p>	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected $>$ $\frac{1}{2}$ RL and $>$ $\frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $>$ RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than \pm 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD \leq 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 6

DoD Specific QC Control Limits

QSM Tables G-4 and G-5

DoD strongly believes that it is important for laboratories to maintain their own in-house LCS limits. These in-house limits must be consistent with (i.e., within) the DoD limits (project-specific, if available; otherwise the following LCS-CLs). The laboratory in-house limits shall be calculated from the laboratory's historical LCS data in accordance with a documented procedure (e.g., SOP) that is consistent with good laboratory practice. That document must describe the process for establishing and maintaining LCS limits and the use of control charts.

The laboratory in-house limits are to be used for several purposes:

- Laboratories are expected to utilize their in-house limits as part of their quality control system, and to evaluate trends and monitor and improve performance.
- When a laboratory's in-house limits are outside the DoD control limits (upper and/or lower), they must report their in-house limits in the laboratory report (see Appendix E) even if the LCS associated with the batch fell within the DoD limits. Using this information, DoD will be able to determine how laboratory performance affects the quality of the environmental data.
- DoD may review the laboratory in-house limits and associated trends, as reflected in control charts, to determine whether the laboratory's overall performance is acceptable. If deemed unacceptable, this can allow DoD to decide not to use the laboratory again until substantial improvement has occurred.

**Table G-4. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260
Water Matrix²**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	105	8	80	130	75	135
1,1,1-Trichloroethane	100	11	65	130	55	145
1,1,2,2-Tetrachloroethane	96	11	65	130	55	140
1,1,2-Trichloroethane	100	8	75	125	65	135
1,1-Dichloroethane	101	11	70	135	60	145
1,1-Dichloroethene	99	10	70	130	55	140
1,1-Dichloropropene	102	10	75	130	65	140
1,2,3-Trichlorobenzene	99	14	55	140	45	155
1,2,3-Trichloropropane	98	9	75	125	65	130
1,2,4-Trichlorobenzene	100	11	65	135	55	145
1,2,4-Trimethylbenzene	103	10	75	130	65	140
1,2-Dibromo-3-chloropropane	91	14	50	130	35	145
1,2-Dibromoethane	100	7	80	120	75	125
1,2-Dichlorobenzene	96	9	70	120	60	130
1,2-Dichloroethane	100	10	70	130	60	140
1,2-Dichloropropane	100	8	75	125	65	135
1,3,5-Trimethylbenzene	102	10	75	130	65	140
1,3-Dichlorobenzene	100	8	75	125	65	130
1,3-Dichloropropane	100	9	75	125	65	135
1,4-Dichlorobenzene	99	8	75	125	65	130
2,2-Dichloropropane	103	11	70	135	60	150
2-Butanone	91	20	30	150	10	170

² A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Total Xylene. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section G.5 and for surrogate compounds in section G.6.

Table G-4. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260
Water Matrix² (continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
2-Chlorotoluene	100	9	75	125	65	135
2-Hexanone	92	12	55	130	45	140
4-Chlorotoluene	101	9	75	130	65	135
4-Methyl-2-pentanone	96	13	60	135	45	145
Acetone	91	17	40	140	20	160
Benzene	102	7	80	120	75	130
Bromobenzene	100	8	75	125	70	130
Bromochloromethane	97	11	65	130	55	140
Bromodichloromethane	98	8	75	120	70	130
Bromoform	99	10	70	130	60	140
Bromomethane	88	19	30	145	10	165
Carbon disulfide	100	21	35	160	15	185
Carbon tetrachloride	102	12	65	140	55	150
Chlorobenzene	102	7	80	120	75	130
Chlorodibromomethane	96	13	60	135	45	145
Chloroethane	99	12	60	135	50	145
Chloroform	100	12	65	135	50	150
Chloromethane	83	15	40	125	25	140
cis-1,2-Dichloroethene	99	9	70	125	60	135
cis-1,3-Dichloropropene	100	10	70	130	60	140
Dibromomethane	101	8	75	125	65	135
Dichlorodifluoromethane	93	21	30	155	10	175
Ethylbenzene	100	9	75	125	65	135
Hexachlorobutadiene	97	15	50	140	35	160
Isopropylbenzene	101	9	75	125	65	135
m,p-Xylene	102	9	75	130	65	135
Methyl tert-butyl ether	94	10	65	125	55	135
Methylene chloride	96	14	55	140	40	155
Naphthalene	96	14	55	140	40	150
n-Butylbenzene	103	11	70	135	55	150
n-Propylbenzene	101	9	70	130	65	140
o-Xylene	100	7	80	120	75	130
p-Isopropyltoluene	102	10	75	130	65	140
sec-Butylbenzene	100	9	70	125	65	135
Styrene	100	11	65	135	55	145
tert-Butylbenzene	99	10	70	130	60	140
Tetrachloroethene	96	18	45	150	25	165
Toluene	100	7	75	120	70	130
trans-1,2-Dichloroethene	99	13	60	140	45	150
trans-1,3-Dichloropropene	98	15	55	140	40	155
Trichloroethene	99	9	70	125	60	135
Trichlorofluoromethane	103	15	60	145	45	160
Vinyl chloride	99	16	50	145	35	165

Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260 Solid Matrix³

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	100	9	75	125	65	135
1,1,1-Trichloroethane	101	11	70	135	55	145
1,1,2,2-Tetrachloroethane	93	13	55	130	40	145
1,1,2-Trichloroethane	95	11	60	125	50	140
1,1-Dichloroethane	99	9	75	125	65	135
1,1-Dichloroethene	100	12	65	135	55	150
1,1-Dichloropropene	102	11	70	135	60	145
1,2,3-Trichlorobenzene	97	12	60	135	50	145
1,2,3-Trichloropropane	97	11	65	130	50	140
1,2,4-Trichlorobenzene	98	11	65	130	55	140
1,2,4-Trimethylbenzene	100	12	65	135	55	145
1,2-Dibromo-3-chloropropane	87	16	40	135	25	150
1,2-Dibromoethane	97	9	70	125	60	135
1,2-Dichlorobenzene	97	7	75	120	65	125
1,2-Dichloroethane	104	11	70	135	60	145
1,2-Dichloropropane	95	8	70	120	65	125
1,3,5-Trimethylbenzene	99	11	65	135	55	145
1,3-Dichlorobenzene	98	9	70	125	65	135
1,3-Dichloropropane	100	8	75	125	70	130
1,4-Dichlorobenzene	98	9	70	125	65	135
2,2-Dichloropropane	101	11	65	135	55	145
2-Butanone	94	22	30	160	10	180
2-Chlorotoluene	98	10	70	130	60	140
2-Hexanone	97	16	45	145	30	160
4-Chlorotoluene	100	9	75	125	65	135
4-Methyl-2-pentanone	97	17	45	145	30	165
Acetone	88	23	20	160	10	180
Benzene	99	9	75	125	65	135
Bromobenzene ⁴	93	9	65	120	55	130
Bromochloromethane	99	9	70	125	60	135
Bromodichloromethane	100	9	70	130	60	135
Bromoform	96	13	55	135	45	150
Bromomethane	95	21	30	160	10	180
Carbon disulfide	103	19	45	160	30	180
Carbon tetrachloride	100	11	65	135	55	145
Chlorobenzene	99	8	75	125	65	130
Chlorodibromomethane	98	11	65	130	55	140
Chloroethane	98	20	40	155	20	175

³ A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Methyl tert-butyl ether and Total Xylene. Sufficient data to perform statistically significant analyses were not received for MTBE during the LCS study. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM; it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section G.5 and for surrogate compounds in section G.6.

⁴ Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

**Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260
Solid Matrix³ (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Chloroform	98	9	70	125	65	135
Chloromethane	90	13	50	130	40	140
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane ⁴	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

**Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270
Water Matrix⁵**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120

⁵ A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

Attachment 7

Additional QA/QC Requirements for MA-DEP



Title: **Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B**

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with BFB	Inter-laboratory consistency and comparability	(1) Criteria listed in Table 4 of SW-846 Method 8260B (the same criteria must be used for all analyses) (2) Every 12 hours	No	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be \leq Reporting Limit (RL) (3) %RSD should be ≤ 15 or "r" should be ≥ 0.99 for all compounds except CCCs which must be ≤ 30 %RSD or "r" ≥ 0.99 (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin.	No	Recalibrate as required by method (1) if any of CCC % RSDs >30 or any of CCC "r" <0.99 or (2) if $>20\%$ of remaining analytes have % RSDs >30 or "r" <0.99 .	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in Environmental Laboratory case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) Percent difference or percent drift must be ≤ 20 for CCCs and should be ≤ 30 for other compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20 , or (2) if %D of $>10\%$ of other analytes >30 .	Report non-conforming compounds in Environmental Laboratory case narrative.
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 samples prior to running samples and after calibration standards (2) Matrix and preservative-specific (e.g., water, MeOH, NaHSO ₄) (3) Target analytes must be $<RL$ except for common laboratory contaminants (such as acetone, methylene chloride, and MEK which must be $<5x$ the RL)	Yes	Locate source of contamination; correct problem; reanalyze method blank.	(1) Report non-conformance in Environmental Laboratory case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the Environmental Laboratory case narrative.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level must be at or near the mid-level (50%) standard (4) Must contain all target analytes (5) Matrix and preservative-specific (e.g., water, MeOH, NaHSO ₄) (6) Laboratory-determined percent recoveries must be between 70 – 130 for target compounds. (7) Can also be used as CCAL	Yes	Recalculate the percent recoveries; Reanalyze the LCS; Locate source of problem; reanalyze associated samples.	(1) Report non-conformances in Environmental Laboratory case narrative. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined recovery ranges routinely exceed the $100 \pm 30\%$ criterion. Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit.



Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
LCS Duplicate	Laboratory Method Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory-determined percent recoveries must be between 70 – 130 for target compounds (6) Matrix and preservative-specific (e.g., water, MeOH, NaHSO ₄). (7) Laboratory-determined Relative Percent Difference (RPD) must be ≤ 25 except for "difficult" (**) analytes which must be ≤ 50.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined RPDs routinely exceed the ≤ 25 criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level - between low (RL) and mid-level (50%) standard (5) Must contain all target analytes. (6) Percent recoveries - between 70 – 130 (7) RPDs should be ≤30 for waters and solids	Yes Only when requested by the data-user	Check LCS; if recoveries acceptable in LCS, narrate non-conformance.	Note exceedances in Environmental Laboratory case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Evaluate surrogate recovery from individual field samples. (2) Minimum of 3 surrogates, at retention times across GC run. (3) Percent recoveries must be between 70-130 for individual surrogate compounds. Laboratory-determined surrogate recovery limits that exceed ± 30% are acceptable for some difficult matrices (wastes, sludges, etc.) with appropriate analytical documentation.	Yes	If one or more surrogates are outside limits, reanalyze sample unless one of the following exceptions applies: (1) obvious interference present (e.g., UCM). (2) for methanol-preserved samples, re-analysis is not required if % moisture >25 and recovery is >10%. (3) if one surrogate exhibits high recovery and target analytes are not detected in sample.	(1) Note exceedances in Environmental Laboratory case narrative. (2) If re-analysis yields similar surrogate non-conformances, the laboratory should report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.



Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards (IS)	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 at retention times across GC run (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard (Section 5.10 of 8260B) (3) Retention times of internal standards must be within ± 30 seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, reanalyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in Environmental Laboratory case narrative. (2) If re-analysis yields similar internal standard non-conformances, the laboratory should report both results. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on IS calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte The IS used for quantitation must be the one nearest the retention time of the subject analyte.	NA	NA	(1) If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes. (2) It is essential that the laboratory clearly document the calculation of analyte concentrations when non-linear calibrations are employed.
General Reporting Issues	NA	(1) The laboratory must only report values \geq the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc) for each analysis must be "reported". (3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit. (3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II A-4. (4) The performance of dilutions must be documented in the Environmental Laboratory case narrative.
GC/MS = Gas Chromatography/Mass Spectrometry BFB = 4-Bromofluorobenzene MS/MSDs = Matrix Spikes/Matrix Spike Duplicates %RSD = Percent Relative Standard Deviation UCM = Unresolved Complex Mixture			"r" = Correlation Coefficient CCC = Calibration Check Compounds RPDs = Relative Percent Differences TIC = Tentatively Identified Compound NA = Not Applicable		
** Potentially "difficult" analytes include: acetone, bromomethane, chloroethane, dichlorodifluoromethane, diethyl ether, dibromochloromethane, hexachlorobutadiene, MEK, 4-methyl-2-pentanone, 1,4-dioxane and trichlorofluoromethane					

Attachment 8

Corrective Action and Documentation Examples

<u>OCCURRENCE</u>	<u>ACTION</u>	<u>DOCUMENTATION</u>
1. Initial calibration does not meet QC criteria.	1. Investigate source of problem, determine if source is an instrument problem or a standard solution problem. If problem is with a single point of the ICAL, reanalyze the bad standard and reevaluate. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	1. Notation in instrument run log book, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification check does not meet QC criteria.	2. Investigate source of problem, determine if source is with ICAL or ICV, is it an instrument problem or a standard solution problem, reanalyze ICV or perform new ICAL.	2. Notation in instrument run log book, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria.	3. Investigate source of problem. If source is instrument, perform instrument maintenance and reanalyze CCV. If CCV still will not pass, repeat the above, or perform new initial calibration. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	3. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
4. GC/MS tune does not meet method criteria.	4. Investigate source of problem, evaluate instrument response to cal gas (PFTBA), when instrument response to PFTBA is improved, re-inject BFB tune.	4. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
5. Method blank contains target		

<p>compound above reporting limit.</p> <p>6. Surrogate standard outside of acceptable range.</p> <p>7. Compound out of acceptance range in laboratory control sample.</p> <p>8. Compound in sample exceeds upper calibration standard concentration.</p>	<p>5. Investigate source of problem. Reanalyze all effected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released with commentary.</p> <p>6. Investigate source of problem. If it is determined to be an instrument problem, reanalyze sample. If it is determined to be a preparation problem, <u>analyze another aliquot of the sample</u>. If it can be determined to be an obvious matrix problem (masking of surrogate by target or non-target compound at significantly greater concentration, excessive hydrocarbons in sample, other knowledge of sample matrix, etc.) the sample may be reanalyzed at dilution to reduce interference or reported with notation in narrative, depending on project objectives.</p> <p>7. Investigate source of problem. If LCS is acceptable per method/SOP specifications, associated sample data can be reported. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP requirements, reanalyze all associated samples. If insufficient sample volume, notify project manager to discuss with client, report initial data if no other sample can be provided.</p>	<p>5. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.</p> <p>6. If only the reanalysis is reported make a notation in the instrument run log. If both sets of data are to be reported, notation in instrument run log, preparation logbooks, commentary on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. If source of problem found to be systematic (bad spike solution, etc). a formal corrective action form must be initiated.</p> <p>7. If LCS is acceptable per method/SOP, flag all compounds out of range on Form 3 of data report, if samples are reanalyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook and commentary in data review checklist to be included in project narrative. If reanalysis cannot be performed due to insufficient sample, commentary in data review checklist to be included in project narrative. If source of problem found to be systematic (bad spike solution, etc), a formal corrective action form must be initiated.</p>
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<p>9. Instrument blank (GC) contains contamination above QC criteria.</p> <p>10. Matrix spike recovery out of QC range.</p> <p>11. Duplicate (or MSD) relative percent difference exceeds QC limit.</p> <p>12. Internal Standard areas exceed QC criteria. (-50% to +100%)</p>	<p>8. Reanalyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running instrument using autosampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be reanalyzed if sufficient volume exists.</p> <p>9. Investigate source of problem, decontaminate purge and trap instrument, reanalyze all effected samples.</p> <p>10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in unspiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, reanalyze spike sample.</p> <p>11. Evaluate problem. If concentration of analyte is</p>	<p>8. Notation in instrument run log book. If initial analysis is reported, flag compound exceeding calibration limit with “E” on data report and commentary on data review checklist to be included in project narrative. If only diluted analysis is to be reported, commentary in data review checklist to be included in project narrative. If both initial and dilution are to be reported, all of the above.</p> <p>9. Notation in instrument run logbook, and if instrument maintenance performed, in instrument maintenance logbook.</p> <p>10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p> <p>11. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p>
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- A. CCV.
- B. QC (blank, LCS)
- C. Samples and MS/MSD.

close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, reanalyze duplicate sample.

12. A. Evaluate problem. Re-analyze CCV. If the CCV does not meet criteria re-analyze the initial calibration and proceed with CCV/QC/samples.

B. Evaluate CCV and QC. As blank and LCS are "interference-free matrix" the IS areas should be within the same limits as the CCV. Evaluate for potential problems. If time allows (data not required on a rush basis), reanalyze QC prior to sample analysis. If insufficient time due to client deadline, data may be reported (as they meet method requirements) but the issue should be noted for the data reviewer and for the client.

C. Evaluate CCV and QC. The IS areas may indicate a potential problem, or matrix interference. If the CCV and batch QC meet criteria, document and report results as matrix interference. In particular, if the recovery of the surrogate standard associated with the IS compound is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery criteria, the IS issue is impacting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias) This requires notation and

12. A. Document in the analytical run log.

B. If the criteria are met after re-analysis, document in run log. If the criteria have not been met and the results of the sample batch are reported, document in the run log and the Corrective Action Logbook. Have the supervisor review situation, initial/date, and include a comment on the data review checklist for the data reviewer and for inclusion in the narrative information to the client.

C. If the CCV and QC meet criteria, document in the run log and on the package checklist for inclusion in the narrative submitted to the client. Note that certain compounds may be potentially high bias or potentially low bias due to IS recoveries outside of range. If QC and samples do not meet criteria and the results are reported, document in the run log, document in the LIMS Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

	<p>communication of the effect to the data reviewer and the client. Based on the severity of the problem, discuss with supervisor, technical director and /or reanalyze effected samples. If results are reported as is, document per 12C.</p>	
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**MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.**

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

SOP No. 30.0003

Rev. 14

	Signature	Date
QA Director:	<u></u>	<u>2/3/10</u>
Lab Director:	<u></u>	<u>2/4/10</u>
Effective Date:	<u>2/11/10</u>	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

Rev. 14

1. Scope and Application

This Standard Operating Procedure describes the procedures that must be followed upon receipt of samples at MITKEM. It describes the procedures to log-in and store samples. It also describes procedures for checking samples out of the storage area for analysis, and for final disposal of samples. Detailed procedures for entering work orders and samples into Mitkem's Laboratory Information Management System (LIMS) are described in a separate SOP Number 20.0003.

2. Personnel Qualifications and Responsibilities

The Sample Custodian should have a two-year degree in environmental science or a related field or have six months of on-the-job experience working with a trained and qualified Sample Custodian.

3. Summary of Procedure

When sample coolers are received, cooler custody seals and temperature are checked. The chain-of-custody forms are signed and the condition of the samples is checked, including any sample custody seals. Each sample is assigned a specific individual sample number. Any discrepancies are documented and resolved. Samples are logged-in to the LIMS following procedures in SOP 20.0003. Labels are generated, and affixed to samples. The pH of aqueous samples (Inorganic only, never VOA) are checked and if need be, either acid or base is added. The addition of preservative (and its lot number) is recorded on the Condition Form. Samples are stored in secure area under documented custody conditions. Certain types of VOC soil samples require special handling, as described in **Section 8.2.2**. Samples are kept at Mitkem for a specific time as called for by contract with the client, and then disposed of in accordance with federal, state and local regulations. The door to the sample receiving room is locked at all times, using a keypad code lock.

4. Sample Preservation, Containers, Handling, and Storage

Samples received at Mitkem are collected by our clients. Mitkem does provide preservatives and sample containers pre-cleaned by bottle suppliers.

Tables 7-1, 7-2 and 7-3 of Mitkem's Quality Assurance Plan (QAP) lists the type of container needed for a particular type of analyte and the proper preservative.

5. Interferences and Potential Problems

Any problem with sample condition including custody seals, breakage, discrepancies in chain of custody or other shipping documentation, cooler temperature, missing samples, etc. must be communicated to the client promptly and resolved. The Sample Custodian or assistant documents the problem and notifies the Mitkem Project Manager, who will then notify the client. For CLP the Project Manager will notify the Sample Management Office (SMO).

6. Equipment and Apparatus

- 6.1 NIST-calibrated temperature "gun".
- 6.2 Condition Forms: Non-CLP, CLP (DC-1).
- 6.3 Orange colored Sample Condition Notification forms.
- 6.4 Narrow range pH paper, capable of recording pH 0- 2.5, and 11-13.
- 6.5 Computer with Accessories to run the LIMS system.
- 6.6 Label printer and labels.

7. Reagents

Reagent grade chemicals shall be used in all tests. Other grades may be used provided the reagent is first verified to be of sufficiently high purity to permit use without lessening the accuracy of the test.

- 7.1 Hydrochloric Acid (HCl), Trace Metal Grade.
- 7.2 Nitric Acid (HNO₃), Trace Metal Grade.
- 7.3 Sodium Hydroxide (NaOH), Certified A.C.S.
- 7.4 Zinc Acetate (CH₃CO₂)₂ Zn 2H₂O, certified.

7.5 Sulfuric Acid (H₂SO₄), Trace Metal Grade.

7.6 Phosphoric Acid (H₃PO₄), Trace Metal Grade.

Reagents are ordered by the inorganic laboratory section, and kept in the sample receiving and bottle prep areas. Zinc Acetate is stored in the volatile laboratory as it is only used with volatile sample bottle prep.

If the reagent needed is in neat form (NaOH or Zinc Acetate) a new bottle may be obtained from the chemical storage area. If you take the next to the last or the last bottle for login dept use, you must inform the inorganic lab supervisor so more can be ordered.

8. Procedure

8.1 Log-In Procedures:

Samples received at Mitkem are received from private courier (FedEx, UPS, etc.) or Mitkem courier or delivery by the clients. All samples received must be accompanied by a chain-of-custody (COC) record.

- 8.1.1 Prior to opening coolers the receiving hood must be turned on, and safety glasses, lab coat, and gloves worn.
- 8.1.2 Select the next sequential red workorder folder with a pre-assigned work order number. Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. For example, 2006 is assigned the alpha character "E" and the 5th set of samples received during 2006 is assigned work order number E0005. Occasionally, a client wishes for a workorder number to be ongoing for several shipments of samples (i.e. EPA). The project manager will notify the sample custodian if additional samples are to be added to an existing workorder (an open Sample Delivery Group or SDG).
- 8.1.3 Before opening coolers, check the outside of the opening of the shipping cooler for the presence of custody seals. Remove any shipping air bills and note the work order number on them. If an EPA cooler is received without an air bill attached, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information. This information should be noted on the Sample Condition Form (See **Attachments 1 and 2**).

NOTE: There are two different types of Sample Condition Forms; one for commercial samples, and one for the US EPA contract samples (see Attachments). Place air bills and all other paperwork for this shipment in the appropriate red workorder folder.

- 8.1.4 Open the cooler underneath the hood and remove the chain-of-custody (COC) form. If a large number of coolers is received, or the cooler is extremely heavy, open it briefly in hallway or on floor and remove the COC only. This is typically located in a plastic zip-lock bag on the top of the samples or taped to the inside of the cooler lid. Write the Mitkem workorder number on the COC. Sign the COC, noting the date and time of receipt of the samples at Mitkem. Proceed with the next steps only after the cooler is under the hood. Look for return air bills to return coolers to the client; these are typically included in EPA projects. If the COC is missing, notify the Mitkem project manager using the Mitkem Sample Condition Notification (printed on orange paper) form immediately. Do not process the samples. Store the samples in the walk-in cooler until the client is contacted and responds.
- 8.1.5 Take the temperature of the contents using the temperature gun or thermometer.

8.1.5.1 Use the Temperature Gun as follows:

- 8.1.5.1.1 Unwrap a sample in the opened cooler. Unwrap enough of the sample in order to take a temperature reading.
- 8.1.5.1.2 Take the temperature by holding the gun no more than two inches from the sample container. Do not aim at the label or cap of the sample. Take several readings on at least 2 samples at opposite ends of the cooler. Record the temperature on the Sample Condition Form and the lower right hand corner of the chain-of-custody or where noted on the Chain-of-Custody. The acceptable temperature range is $4^{\circ} \pm 2^{\circ}\text{C}$ (2° to 6°C). If the temperature is acceptable, move on to **Section 8.1.6**. If the temperature is not within this range, enter the temperature on the Sample Condition Form, circle "yes" to the question regarding the Sample Notification form, and fill out the Sample Notification form (See **Attachment 3**). For CLP projects, if the temperature exceeds 10°C , the project manager will contact SMO for corrective action guidance. Give the entire folder and the Sample Notification form to the project manager. The project manager will contact the client for instructions on how to proceed and return the folder to the sample custodian indicating action to be taken. During the interim, the samples in

question will not be processed, but the closed cooler will be stored in the walk-in cooler.

8.1.5.1.3 Record the Temperature Gun ID on the sample condition form.

8.1.5.2 If a temperature blank is present, use a thermometer as follows:

8.1.5.2.1 Insert the thermometer in the temperature blank.

8.1.5.2.2 Let stand for a minimum of three minutes to as long as five minutes maximum.

8.1.5.2.3 Read the temperature and record it on the Sample Condition Form and in the lower right hand corner of the COC or where noted on the Chain-of-Custody. If the temperature is not within $4^{\circ} \pm 2^{\circ}\text{C}$ (2° to 6°C), proceed as in **section 8.1.4** above. For CLP projects, if the temperature exceeds 10°C , the project manager will contact SMO for corrective action.

8.1.5.3 A calibration check of the thermometer is to be done quarterly or as the thermometer is replaced by comparing it's reading to the NIST thermometer and recording the results in the QA/QC Thermometer Calibration Logbook. See **SOP No. 110.0006** for the calibration procedure.

8.1.5.4 The presence/absence of a temperature blank in the shipment is documented on the Form DC-1. Follow the instructions on the DC-1 Form for contacting SMO, and for documentation in the event a temperature blank is not present.

8.1.6 If the Sample Custodian is not present, the person receiving the samples should perform all steps through **Section 8.1.5.2.3** then store the cooler in the walk-in refrigerator, R1, until the Sample Custodian returns, after removing any VOC samples. Samples for VOC analysis require special handling, as described in **Section 8.2.2**. In general, if not logged-in immediately, VOA vials must be removed from the cooler and stored in refrigerator R2 until logged-in to the LIMS. EPA SOM samples needing temporary storage should be discussed with the SOM project manager. If VOC samples are received in Encore devices (typically contained in silver foil pouches), or are noted as requiring freezing to preserve, they must be removed and stored in the freezer F2. Encore samples must be extruded into pre-weighed VOC vials as soon as possible. Contact one of the following to insure this occurs: VOA Laboratory Supervisor, Sample Custodian, Project Manager, Laboratory Director or QA Director. Leave the paperwork in the red folder on the sample custodian's desk.

- 8.1.7 Once all of the coolers received have been inspected as above, prioritize them for logging into the LIMS with fastest turn-around times and short holding times processed first. The labs should be notified immediately about quick-turn analyses. This can be done either verbally, or by copying the COC and distributing that to the laboratories.
- 8.1.8 Remove and inspect samples from the coolers for the first workorder. Sample containers are removed from the cooler and lined-up on the counter in the same order as listed on the chain of custody form. Check to see that the correct number of samples is present. Check to see if any samples are broken. Check aqueous VOA sample vials for the presence of air bubbles or headspace. If bubbles or headspace are present, a Sample Notification form should be filled out and given to the Project Manager. Check to see that the quantity present is sufficient for the test requested. Make sure the client IDs on the bottles agree with the COC. Check the custody seals, if any, on the sample containers to ensure the all seals are present and intact.
- 8.1.9 For U.S. EPA samples, sign the COC and note the temperature on the bottom right corner of the COC. Place the project number in the top right hand corner of the COC. Record the following information on the Form DC-1.
- Sign DC-1. Note that the person signing should be the person who signed for receipt on the COC. There will be one DC-1 form for each cooler received for a particular case. Only the samples in that particular cooler will be listed on that DC-1 form.
 - EPA Case number from the COC.
 - List sample tag control numbers and compare these with the COC records.
 - Document whether or not these numbers agree.
 - If Sample tag numbers are not listed on the COC form, record this fact.
 - Record the custody seal numbers if present.
 - Write the work order number on the air bill and place it in the red project folder. Record the name of the courier and the air bill number on Form DC-1.
 - Date of receipt.
 - Time of receipt.
 - EPA sample numbers.
 - SDG-Final Sample must be documented on EPA TR/COC.
 - SDG (Sample Delivery Group) number. The SDG is the lowest alphanumeric sample on the first shipment and remains the same until the work order is closed.
 - EPA sample numbers.
 - Assigned Lab ID numbers. Note that with EPA projects, shipments may be received over several days to be added to the same work order, up to twenty field samples, not including PE samples. The SDG number stays the same, but the sample ID numbers run consecutively.

- Whether samples were delivered by hand or not.
- Any problems and discrepancies in “remarks” box.
- The cooler temperature is entered in item #10 of the Form DC-1.

Note from the COC whether the work order is closed, or if more samples are expected to be added (work order/SDG is still open). EPA projects are closed automatically after one week from the date of receipt, for cases with a turnaround time of 14 or 21 days and three days for samples with a turnaround time of 7 days, of the first shipment or sooner if the client checks the box that the project is closed.

Discrepancies in EPA shipments should be reported to the Project Manager directly. Any irregularities in the above information should be documented and the client contacted by either Project Manager or the Sample Custodian. Until an answer is received from the client, samples should be stored in the walk-in cooler. Volatiles samples must be stored in R/F2.

If there are any discrepancies, note them on the Sample Condition Notification Form and give the form and the workorder folder to the project manager. Samples with issues will not be processed further, but stored in the walk-in cooler until a resolution has been reached with the client. For CLP (EPA) samples, the Sample Management Office (SMO) will be contacted for corrective action. Communication of the corrective action will be placed in the red project folder.

- 8.1.10 Log the workorder and samples into the Laboratory Information Management System (LIMS) following procedures described in SOP 20.0003. Labels are generated and attached to each sample container. In the case of pre-preserved samples in VOA vials (sodium bisulfate, DI water and/or methanol), be sure the Mitkem sample ID label does not cover the tare weight of the vials.
- 8.1.11 Have the project manager or a second person check the Lab IDs on the labels vs. the client IDs on the COC to confirm the bottles have been properly labeled. Ensure the reviewer initials the proper line of the Sample Condition Form or DC-1 form.
- 8.1.12 Enter the workorder number, date received, and client name in the Sample Receipt and Tracking Logbook (**Attachment 6**). Occasionally, a client wishes for a project number to be ongoing for several shipments of samples (i.e. EPA). In such cases the date of arrival of each subsequent shipment should be noted for the original work order number.
- 8.1.13 Inorganic preserved samples must now be tested for pH as follows:

For metals, wet chemistry, etc., pour a small amount of sample over a strip of pH paper that is suspended over the mouth of a waste container. The pH of the acidified samples is taken by using the narrow range pH strips which measures

pH 0 to 2.5. The sample pH should be less than two, as indicated by the color on the pH indicator package. The pH of the basic samples is taken by using the narrow range pH strips which measures pH 11.0 – 13.0. The basic samples should have a pH 12 or greater, as indicated by the color of the pH indicator package. (See **Attachment 4** for instructions on the use of the pH paper.) Acidified samples must have a pH of less than 2. Any pH adjustments that need to be made are recorded on the Sample Condition Form for the workorder. Record the lot number and ID of the reagent used and the approximate volume used to adjust the pH. For metals samples requiring acidification, alert the inorganic laboratory to allow 24 hours before metals preparation to allow enough time for the acids to dissolve any metals that adsorb to container walls.

- 8.1.14 Some samples (i.e. fecal coliform bacteria) which require subcontracting must be done so immediately as the analysis has an extremely short hold time. A list of analyses typically subcontracted can be printed from the LIMS Omega system. An Outside Services Purchase Order is also generated from the LIMS system when the batch of samples to subcontract is created.
- 8.1.15 Receiving personnel make one copy of the sub contract COC. This COC accompanies the samples and is signed by the laboratory that will perform the analysis. A copy of the COC is placed into the red workorder folder and one copy is sent to Accounting.

8.2 Storage and Location of Samples:

8.2.1 Semivolatile Organic, Pesticides/PCBs, and Wet Chemistry Samples:

Samples are stored in the main walk-in refrigerator (R1) in the sample receipt room at $4^{\circ} \pm 2^{\circ}\text{C}$. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook. The person putting the samples into the walk-in must put their initials in the logbook beside the location entries.

8.2.2 Volatile Organic Samples:

Samples for VOA are stored separately from all other samples, in refrigerators or freezers dedicated for only this purpose. These refrigerators and freezers are located in the VOA Laboratory. A soil sample for VOA may contain one or more of the sample types listed below. Care must be taken to insure each sample vial type receives the proper handling. Soil samples for volatiles analysis must have special handling performed by the VOA lab staff.

A storage blank must accompany all VOA samples requiring EPA/CLP or New York State ASP analyses when placed in the VOA refrigerators. The storage blanks are prepared in the volatiles department using the VOA Type I water.

The 40ml vial is filled so there is **no** headspace, capped immediately and tightly, and labeled with the date prepared and any preservative added. The storage blanks for unpreserved low/medium soil samples are prepared by placing approximately 5 g of inert sand in dry closed-system purge-and-trap vials. These inert sand storage blanks will be stored in the same freezer with unpreserved low/medium soil samples.

- 8.2.2.1 Samples suspected of containing high concentrations of volatile organics must be stored in a separate VOA refrigerator designated for such samples. This isolates these high concentration samples from other samples, and helps to control cross-contamination.
- 8.2.2.2 Soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial) should be stored in a VOA freezer (containing no standards or sample extracts). These vials are to be weighed by VOA lab staff prior to analysis.
- 8.2.2.3 Soil samples that contain up to 5 mL of water. These vials are also to be stored in a VOA freezer. These vials are to be weighed by VOA lab staff prior to analysis.
- 8.2.2.4 Soil samples that contain 5mL of water containing a sodium bisulfate preservative. These vials are also to be held in a VOA refrigerator (specifically for EPA CLP samples). Samples for Method 8260 analysis may be held in a VOA refrigerator. These vials are to be weighed by VOA lab staff prior to analysis.
- 8.2.2.5 Soil samples received in the Encores (typically contained in silver foil pouches), the samples must be extruded into tared, dry closed-system purge-and-trap vials by the VOA lab staff, and then re-weighed to obtain the final sample weight. This should happen immediately (for EPA CLP samples), but no longer than 48hours of sample collection. The vials are then to be placed into a VOA freezer.
- 8.2.2.6 Soil samples may also arrive as methanol preserved samples. These samples do not require freezing, but they shall be held in a VOA refrigerator to be weighed by VOA lab staff prior to analysis. If possible, check that the methanol covers the soil samples within the vial. If the methanol is insufficient, note this on the Sample Condition Form and relay the information to the VOA lab.
- 8.2.2.7 Unpreserved VOA soil samples should be stored in a VOA freezer (EPA CLP samples) or a VOA refrigerator (for Method 8260 samples) until the time of analysis.

8.2.2.8 Water samples are stored in VOA refrigerators. Trace Volatile samples for the EPA/CLP SOM01.2 contract must be stored in a separate refrigerator from all other samples. At present, this has been designated as **R13 and R4**.

8.2.2.9 Log the samples into the VOA lab. Enter the workorder and other pertinent information in the Volatile Receiving Logbook (See **Attachment 7**) and sign off that the samples have been relinquished. Different sample preservation types that result in different storage designations require separate line entries in the logbook (ex: SOM methanol extracts into refrigerator on one line, associated unpreserved soils into freezer on another line) If the samples are ever removed from VOA they must be signed out of the VOA lab as well.

8.2.3 Metals Samples:

Aqueous preserved samples for metals analyses that do not require refrigeration are stored on the rack in the sample receipt area.

8.2.3.1 ISM water and soil samples require refrigeration at $4^{\circ} \pm 2^{\circ}\text{C}$ and must be stored in the walk-in. Cyanide aqueous samples must also be protected from light. Only aqueous cyanide samples are required to remain stored in refrigeration after distillation. All other matrices and ISM test methods may be stored at room temperature after preparation, until disposal. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook. The person putting the samples onto the rack must put their initials in the logbook beside the location entries.

Standards must be stored in separate freezers or refrigerators isolated from samples and sample extracts.

8.3 Storage and Location of Extracts:

Extracts for semivolatile analyses must be stored, shielded from light, at $4 \pm 2^{\circ}\text{C}$ in a separate refrigerator stored either in the laboratory or in Unit 3. Extracts for Pest/PCB are stored in refrigerators in the same manner.

Extracts must be stored separately from standards.

Extracts for semivolatile and Pest/PCB must be stored until the proper time to dispose of them per client approval. Mitkem normally retains sample extracts for a period of three months from the date of delivery of the final report to the client

USEPA CLP and New York State ASP extracts must be stored for 365 days after delivery of a complete data package to the client.

8.4 Temperature Monitoring:

The temperature of the storage areas must be monitored on a daily basis, including weekends and holidays. This temperature is recorded electronically using temperature probes that are affixed inside all refrigerator and freezer units. Specific employees are notified of temperatures outside of the acceptance ranges by email so that corrective action can be initiated. If any refrigerator/freezer fails, first investigate the problem. Was the door ajar? Is something preventing the door from shutting fully? If the situation can be rectified, do so and check the temperature again after a sufficient period of time. Adjust the temperature control knob in the unit if other attempts have not fixed the problem and continue to monitor the temperature. All adjustments and resolutions must be entered into the Temperature Probe Program "Event" log. When the temperature meets criteria, document that the refrigerator/freezer has been "*returned to control*". If there is no obvious issue with the refrigerator/freezer, or the refrigerator does not maintain the correct temperature, the QA Director or Lab Director must be notified immediately to correct the problem. Fill out a report in the LIMS Omega Corrective Action Report. Acceptable temperature ranges are 2° – 6°C for refrigerators and –10°C to –20°C for freezers. The temperatures are recorded electronically multiple times daily. See Mitkem SOP 80.0020, Temperature Monitoring Systems.

8.5 Tracking of Samples and Extracts:

8.5.1 Sample Log-out:

When samples are taken from their storage areas for extraction, they must be signed out using the appropriate Sample Receipt Logbook. During 2010, Mitkem will be transitioning from the existing system to the use of bar-coded scanners to document chain of custody of samples from the storage area into the laboratories for preparation and/or analysis. Presently, the log-out procedure is a two-person procedure that requires samples to be relinquished by either the Sample Custodian or Authorized Laboratory Personnel

The analyst retrieves the samples from the walk-in cooler and the Custodian or Authorized Laboratory Personnel checks the sample numbers. The Mitkem sample numbers as well as the bottle # are recorded (i.e. E0140-02B#2). Both people initial and date the Receiving Logbook in the appropriate places. When the sample is logged-out by an analyst, it is considered to be in the custody of that analyst until he/she returns the sample to its storage area.

Upon the sample's return to the storage area the samples are signed into Receiving using the same procedure. This time the analyst relinquishes the samples and the Custodian or Authorized Laboratory Personnel checks the

sample numbers to be returned. Each person signs and dates in the appropriate places in the Receiving Logbook.

Authorized laboratory personnel are employees who are knowledgeable in regard to the receipt and log-in of samples. A list of Authorized Laboratory Personnel is posted on the Receiving bulletin board. (See **Attachment 5**).

8.5.2 Sample Extraction:

If a sample is signed out for extraction, the sample is logged into the lab by entering the Lab ID into the appropriate extraction logbook. After extraction, any remaining sample volume is returned to the storage area and signed back into the Receiving area for storage as noted in **Section 8.5.1.1**.

When working with samples in the preparation laboratories, the following procedures must be adhered to:

- ° All activities performed on the sample must be recorded.
- ° Entries in the logbooks must be signed and dated (mm/dd/yyyy).
- ° All entries must be made in ink.
- ° Corrections and additions must be made by drawing a single line through the errors, entering the correct information, and initializing and dating the new information.
- ° Unused portions of the logbooks must be lined-out or "Z-ed" out.
- ° Logbook entries must be made in chronological order.
- ° For US EPA/CLP samples, entries are recorded for only one SDG on a page, except in the event where SDGs share quality control samples.
- ° Information inserted in the logbooks must be permanently affixed, signed, and dated across the insert.
- ° Copies of all pertinent logbook pages must be placed in the workorder file.
- ° Proper subsampling techniques can be located in the Subsampling SOP, 110.0039.

8.5.3 Extracts/Digestates:

The organic sample extracts, once completed by the preparation laboratory, are transferred to the appropriate instrumentation lab and signed into the extract transfer logbook. The analyst will sign for the extracts and enter storage location information. Inorganic metals and mercury digestates are transferred to the instrumentation lab. The analyst signs off on the preparation logbook at time of transfer.

When the time period for organic extract storage has expired, extracts may be disposed of in the same manner as would used/expired standards. CLP

extracts must be stored for one year. The transfer and disposal for these extracts must be documented in the Extract Disposal Logbook.

When the time period for inorganic digestate storage has expired, digestates are disposed in the same manner as would used/expired inorganic standards.

See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the actual disposal of above extracts/digestates.

8.5.4 Sample Disposal:

The unused portion of sample is returned to the Receiving area for long term storage. Such portions must be stored for a certain length of time as required by client contract and then properly disposed.

All unused portions of EPA SOM and ISM samples must be protected from light and refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (where appropriate for sample type) from the time of receipt until 60 days after the delivery of a complete, reconciled data package. Empty containers must also be retained for a period of 60 days after data submission, but do not require refrigeration. Presently empty containers are held in the storage hallway on specified shelves, in Unit 3. After 60 days, the samples and containers may be disposed of in a manner that complies with all applicable regulations.

Unused sample is disposed with the use of a licensed hazardous waste contractor. Routinely, one of the sample custodians runs the LIMS Omega Disposal program to produce a list of samples ready for disposal. See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the different waste streams and disposal options.

All soil samples received from outside of the 48 contiguous United States must be disposed of following special procedures outlined by the USDA, as detailed in Mitkem SOP No. 30.0024 Sample and Waste Disposal section 8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

- 10.1 The Sample Custodian will calibrate the temperature gun using an NIST calibrated thermometer on a quarterly basis. The correction factor will be noted on the temperature gun and recorded in the NIST Thermometer Calibration Logbook. The serial number of the NIST thermometer will also be recorded in the logbook.

- 10.2 On a daily basis, the Project Managers will review that the Sample Condition Forms are filled out correctly.
- 10.3 Multiple times daily, the temperatures of the refrigerators and freezers will be checked and the temperatures recorded electronically. Deviations will be noted in the electronic logbook and appropriate personnel notified for corrective action. If after 36 hours the temperature problem cannot be corrected with manual adjustments, the refrigerator or freezer will be replaced.
- 10.4 The Hazardous Waste Inspection Logbook will be completed weekly, after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11. Data Validation and Reporting

A copy of the chain of custody and Sample Condition Notification form accompanies the data reports to the client. Also any communication records are to be included in the data package (telephone conversations, e-mail communication, etc.).

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range. Notification reports (orange sheets) are generated in the event of an out-of-control situation occurs at sample log-in that cannot be corrected. Project Managers contact the client in order to ensure what type, if any, corrective action needs to be taken. Other corrective actions concerning the Receiving Area are recorded using the LIMS Corrective Action Report.

13. Health and Safety

The sample custodian must wear a lab coat, safety glasses, and gloves when handling samples and preservative chemicals. Samples coolers should be opened under a hood. All samples should be handled as though they are potentially hazardous. The outside of sample containers should also be handled as though they are contaminated. Any broken sample containers must be handled very carefully to prevent exposure to the sample.

14. Pollution Prevention, Waste Management, and Abbreviations

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

US EPA CLP SOW SOM01.2
US EPA CLP SOW ILM05.4

US EPA CLP SOW ISM01.2
US ACOE Shell for Chemical Analytical Requirements, Appendix H
Mitkem Quality Assurance Plan

Attachments:

1. **Attachment 1:** Mitkem Sample Condition Form.
2. **Attachment 2:** EPA Sample Condition Form (DC-1).
3. **Attachment 3:** Sample Condition Notification Form.
4. **Attachment 4:** Instructions for the use of pH paper.
5. **Attachment 5:** Example Authorized Personnel list.
6. **Attachment 6:** Sample Receipt and Tracking Logbook.
7. **Attachment 7:** VOA Receiving Logbook

Attachment 1
Mitkem Sample Condition Form

Attachment 2

EPA Sample Condition Form (DC-1)

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name Mitkem Laboratories			Page <u>21</u> of <u>32</u>		
Received By (Print Name)			Log-in Date		
Received By (Signature)					
Case Number		Sample Delivery Group No.		Mod. Ref. No.	
Remarks: (1) Please see associated sample/extract transfer logbook pages submitted with this data package.		Corresponding			Remarks: Condition of Sample Shipment, etc.
		EPA Sample #	Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of- Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction (1) TVOA/VOA	Fraction (1) SVOA/Pest/Aroc				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

Attachment 3
Sample Condition Notification Form

Sample Condition Notification

Mitkem Project#: _____

Date of Receipt: _____

Client: _____

Received By: _____

Client project #/name: _____

Unusual Occurance Description:

Client Contacted:

Contacted via: Phone/Fax/E-mail

Date: _____ Time: _____

Contacted By: _____

Name of person contacted: _____

Client Response:

Responded via: Phone/Fax/E-mail

Date: _____

Name of person responding: _____

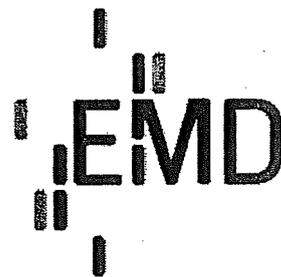
Responding to: _____

Mitkem Action Taken:

Attachment 4

Instructions for use of pH paper

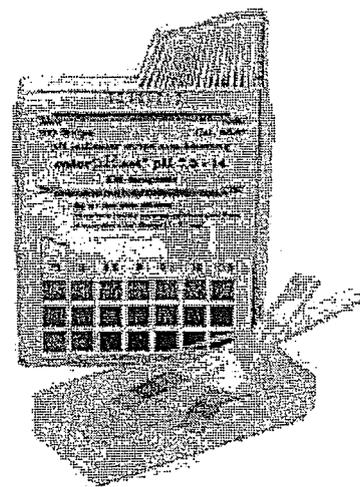
General Procedure for the Use of colorpHast® pH Test Strips



1. Remove a strip from the box.
2. Immerse the reaction zone of the strip into the solution for approximately 30 seconds to 1 minute, allowing enough time for the reaction to take place.
3. Remove the strip from the solution, wiping the strip along the edge of the vessel to remove excess liquid from the strip.
4. Compare the strip's reaction zones to the color chart on the box while holding the strip to the top of the box and the reaction zones to the bottom of the box.
5. Record the results of the closest matching color to the strip.

• *For additional application or product information, ordering information or other technical questions, please call our Technical Service Department at:*

1-800-222-0342 or 1-856-423-6300



Attachment 5

Example Authorized Personnel list

Sample log-in/out: Authorized Personnel

Name	Dept	Initials
Anderson, Courtney	Inorganics	CJA
Appolonia, Gary	Inorganics	GMD
Badura, Karolina	PEST/PCB	KB
Cardoso, Antonio	Oprep	AC
Datta, Avijit	Receiving	AED
Ding, Yihai	Mitkem	YD
Huntley, Agnes	Project Management	ARH
Kaczorowski, Przemyslaw	Oprep	PK
Lawler, Edward	Project Management	EL
Lawler, Sharyn	QA/QC	SBL
Luo, Wei	VOA	WL
Lucas, Derek	PEST/PCB	DL
Maczewska, Beata	SVOA	BM
Marquis, Ashley	VOA	ALM
Thomson, Cassandra	Inorganics	CT
Mosher, Cassie	SVOA	CLM
Montmarquet, Timothy	Mitkem	TM
Ng, Shirley	Project Management	SN
Rosadzinski, Tomasz	Oprep	TR
Scarpaci, Matthew	SVOA	MMS
Sawyer, Tom	Inorganics	TS
Smart, Dawne	Inorganics	DES
Wetherbee, John	Inorganics	JRW
Veilleux, Brian	Oprep	BV
Wieczorek, Agnieszka	PEST/PCB	AW
Zharkova, Sofya	VOA	SZ
Zhao-Anderson, Huiyan	Inorganics	HZA

Most common personnel using log-in/out

Attachment 6

Sample Receiving and Tracking Logbook

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc.

Sample Receiving Logbook

Workorder No. _____

Client Name: _____

Date Recv'd _____ Sample #s _____ Storage Locations: _____

Date Recv'd _____ Sample #s _____ Storage Locations: _____

Date Recv'd _____ Sample #s _____ Storage Locations: _____

Date Recv'd _____ Sample #s _____ Storage Locations: _____

Date Recv'd _____ Sample #s _____ Storage Locations: _____

OUT				IN			
Relinquished By		Received By		Relinquished By		Received By	
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							
Date:	Init:	Date:	Init:	Date:	Init:	Date:	Init:
Samp. #s							

Comments: _____

Please record analyst's initials, date, and sample #s removed. Add any comments if necessary (broken bottles, empty jars, etc.) Include the abbreviated name of the test to be performed, ie: SVOA, PCB...near the "samp. #s". Include bottle or jar number when more than one.

Reviewed: _____

Attachment 7

Volatiles Receiving Logbook

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

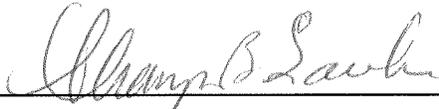
SOP No. 30.0024

Rev. 8

Signature

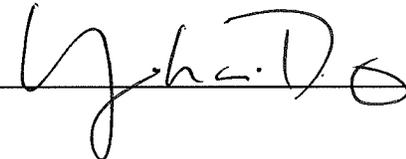
Date

QA Director:



10/17/08

Lab Director:



10/21/08

MITKEM LABORATORIES
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP 30.0024 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the procedures for the disposal of the unused portion of samples submitted for analysis, as well as solid and aqueous wastes and sample extracts to minimize the amount of waste in the lab and organize the manner in which it is handled.

2. Personnel Qualifications and Responsibilities

Personnel handling samples must be aware of the potential dangers posed by the samples, and handle them properly to minimize this danger. Personnel handling samples must also maintain the documented chain of custody of the samples from the time they are received at Mitkem until they become laboratory waste. Laboratory waste must be handled in a safe manner to minimize any exposure to this material.

3. Summary of Procedure

Waste stream manifest profiles have been identified by Mitkem and the waste-disposal company. These profiles dictate how to dispose of each individual waste, as described in this SOP. If any question arises regarding the disposal or classification of waste, the Waste Disposal Technician, or senior laboratory management should be consulted.

Once sample analyses are complete and a final report has been issued to the customer, samples are stored for a specified length of time before disposal. This timeframe is usually dictated by the client. Strict chain of custody documentation is maintained for these samples until samples are disposed into one of several waste streams. This waste is removed from Mitkem by a licensed waste-hauler for final disposal.

Safety equipment will be worn prior to and during any waste disposal. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Waste drums. Polyethylene drums are used for liquids (acids or basic/spent CN solutions) and steel open top drums are used for waste solid materials.

Burp-free Ultra Funnel, various sizes, polyethylene funnel used to allow safe pouring of liquid wastes into drums. Product is vented and has locking lid.

Spill kits

Drum Pump

Drum Lift

Satellite Nalgene storage containers with lidded funnels for interim liquid waste storage in Wet Chem, Volatiles and Metals Labs.

Satellite storage containers (red metal Justrite Oily waste Can ,10G capacity) for interim solids storage in Organic Prep, Semivolatiles and Volatiles Labs.

Satellite storage container (Justrite Safety Disposal Can, 2G capacity) for solvent waste in Volatile Lab.

7. Reagents

Not applicable.

8. Procedure

8.1 Classification of Waste:

Laboratory waste products fall under one of the following categories:

8.1.1 Methylene Chloride, Methanol, Water:

Waste solvents are emptied into a solvent drum located in the organic sample preparation laboratory. This waste consists primarily of methylene

chloride with lesser portions of methanol and water. Trace amounts of other solvents may also be included from time to time. Due to the high proportion of methylene chloride in the waste, the material is flammable, and is classified as D001-type waste. Whenever a new drum is started, a Hazardous Waste label is attached to the outside.

When the drums are full the waste drum is taken to the Hazardous Waste storage shed. An accumulation date is added to the Hazardous Waste label on the drum.

Empty solvent bottles are evaporated to dryness and discarded into the general solid waste dumpster (garbage). Glass containers that present a risk of injury should be discarded into a broken glass container for disposal.

Any lab apparatus, gloves, paper towels, etc. exposed to methylene chloride must be discarded into the red solid waste drums in the Organic Prep Lab. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.2 Acids, Water:

Acid waste consists of approximately 98% water and 2% acid. This waste stream also includes the remainders of spent or expired metals standards, digested samples, TCLP and SPLP leachates and undigested aqueous samples preserved with acid. This waste is emptied into polyethylene acid waste drums in the hazardous waste area. Acidic waste from the Lachat analysis of nitrate/nitrite or chlorides are stored temporarily in Unit 3 in a small waste container equipped with a closing safety funnel. The container is routinely emptied into this same wastestream. A Hazardous Waste label is affixed to the side of the drum and an accumulation date is recorded on it the first time the drum is started. This waste is classified as D002 type waste. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.3 Scintillation Vials, Methylene Chloride, Hexane:

This waste stream consists of Organic analysis vials used for GC and GC/MS analyses. Spent or expired organic standards in solvent are also included in this wastestream. The vials are disposed of unopened. Vials are initially disposed in the GC Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone:

This waste stream contains the remainders of solid and soil samples. This waste stream also includes laboratory solid waste that has been stored in the red metal drums in the Organic Prep Lab. When full the laboratory red metal containers are taken to the Hazardous Waste shed and the contents dumped into the waste solids drum. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and is later removed by the Waste Hauler within 90 days of the accumulation date. This waste has no RCRA Characterization code.

Any solid matrix samples received from outside of the 48 contiguous United States, or from any other area identified by the client as a US Department of Agriculture Soil Quarantine Area must have special disposal in order to kill any quarantined organism. Samples identified as such, along with their entire container including jar cover, are placed in a disposable aluminum pan and placed in the kiln (in Unit 3) and heated to 400 degrees for 30 minutes. After the pan has cooled, the sample(s) and aluminum pan(s) are disposed in the waste soil/solid drum. See **Attachment 1** for incineration procedures.

8.1.5 Glass Vials, Methanol, Water, Soil:

This waste stream consists of primarily 40-ml organic analysis vials used for VOA analyses. The vials are disposed of unopened. Vials are initially disposed in the VOA Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.6 Spent Carbon with Methylene Chloride:

This waste stream contains the remainders of Mitkem's discharge. Discharge is run through tanks of carbon material as a means of removing methylene chloride. When the tanks are fully deactivated, the carbon is removed and dumped into a steel drum. The tanks are then filled with activated carbon. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and the drum is later removed by the Waste Hauler within 90 days of the accumulation date. This waste stream is not associated with any RCRA Characterization code.

8.1.7 Electronic Device with Beryllium Oxide:

This waste stream contains the power tube of an ICP spectrophotometer. Because of beryllium in the tip of the tube, it cannot be disposed of with

ordinary trash. The tube is placed in a sealed cardboard box and a Hazardous Waste label with an accumulation date is affixed to the outside of the box. The box is later removed by the Waste Hauler. This waste stream is not associated with any RCRA Characterization code.

8.1.8 COD Vials:

This waste stream consists of glass tubes that contain a mixture of mercuric sulfate, silver sulfate, sulfuric acid, and chromic acid. The tubes are used for COD analyses and are disposed unopened in their entirety. Vials are initially disposed in the Wet Chemistry Laboratory in a covered cardboard box with a Hazardous Waste Label. When this box is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D002, D009, D011 waste.

8.1.9 Lab Pack Waste:

From time to time, waste or unused chemicals (non-solvents) will require disposal. These materials are segregated and placed in a lab pack container. An inventory of materials is maintained for the lab pack drum. The materials are stored in the laboratory in a closed cardboard box with a Hazardous Waste label attached. Consult the waste hauling company when sufficient volume of material is present in the laboratory to initiate a lab pack.

8.1.10 PCB Waste:

Any PCB-containing material (at a concentration of greater than 50 ppm) must be segregated and disposed according to PCB-specific procedures. This material would generally consist of any oil sample containing over 50 ug/g (ppm) of PCB. Samples containing this high concentration of total PCB will be identified by the GC Laboratory Manager or the Laboratory Director. The container for this sample will be prominently labeled "for return to client" when the holding period (as identified in **Section 8.2.1** below) is reached. The Mitkem project manager will be notified, as will the client, with a notation in the report Project Narrative or Cover Letter that the sample will be returned.

8.1.11 Water/Sodium Hydroxide/Pyridine:

This waste stream consists of sulfide and cyanide digestates and spent standards. Pyridine waste from the analysis of Cyanide (Lachat), and basic pH Ion Chromatography (IC1) wastes are stored temporarily in Unit 3 in several small waste containers equipped with closing safety funnels.

The containers are routinely emptied into a polyethylene Basic Waste drum in the hazardous waste area. This waste is classified as D002, D038 waste

8.2 Procedure for Disposal of Sample Remainers:

8.2.1 Determination of Disposal Date:

Samples are held for a period of time following receipt at the laboratory or release of the final report to the client. Mitkem's policy is to hold samples for a specified length of time. On a case-by-case basis, arrangements may be made between the client and Mitkem through discussion with the Mitkem Project Manager. This agreement is then saved in LIMS in the individual Client Project. Default values are used otherwise. To review the Sample Hold Times and Disposal Options on a particular workorder, go to the Report Option at the Workorder Main screen.

For CLP, samples are held for 60 days after delivery of the reconciled data package. Sample extracts must be stored at 4°C until 365 days after delivery of a complete, reconciled data package to EPA.

8.2.2. Documentation of Sample Disposal:

When the specified hold time has passed since the receipt date, the samples can be disposed. Sample receiving personnel select sample remainders from their storage locations and remove these to a designated location outside of the sample receiving area for disposal by Sample Receiving or laboratory personnel. Documentation that sample remainders have been disposed is maintained in the Mitkem Omega LIMS. When samples are removed from storage for disposal the person removing them enters the disposal date and their initials into LIMS.

8.2.3. Final Disposal and Record keeping:

All samples are to be disposed of according to **Section 8.1.**

Once waste drums are full, approximately bi-monthly, the waste hauling company is called to schedule a pick-up. Mitkem currently uses Univar to arrange for disposal of laboratory waste. Univar will remove the waste from the Mitkem storage area, and arrange with Pollution Control Industries (PCI) in Michigan for ultimate disposal of the waste material. Waste manifests are initiated by the hauler, signed by the QA Director or his designee when drums are loaded on the hauler's truck, signed and mailed back to Mitkem following final disposal. Manifests are filed

according to date in the QA office. Mitkem's Generator Number is **RI500009621**.

9 Data Reduction and Calculations

Not applicable.

10 Quality Assurance/Quality Control

The Hazardous Waste Inspection Logbook will be completed weekly after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11 Data Validation and Reporting

After sample disposal, the disposal dates are entered into the LIMS for each workorder. In addition, the samples' storage location is changed to "disposed".

12 Corrective Action Procedures

Corrective actions concerning sample or waste disposal are recorded using the Omega LIMS Corrective Action Report under the Receiving Department or another lab area involved in the issue. Corrective actions should be implemented in the event of an out-of-control situation involving sample or waste disposal that cannot be easily corrected. The sample custodian should contact the Safety team, Hazardous Waste Coordinator or Lab Director for help determining what corrective action needs to be taken.

13 Health and Safety

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the Mitkem Safety Manual. Bottle or container labels also provide important information that must be noted. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

All laboratory employees who either sign a Hazardous Waste manifest or handle hazardous waste directly must attend an annual RCRA training class. At this time Summit Technologies of New Hampshire provides the necessary on-site training for Mitkem's laboratory personnel.

14 Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15 References

None.

Attachments:

- 1. Attachment 1:** Instructions for the Kiln.

Attachment 1

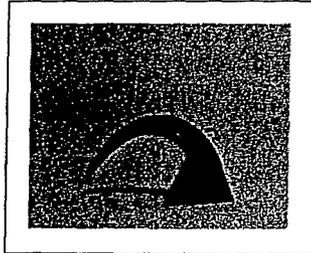
Instructions for the Kiln

FIRING CONTROLS

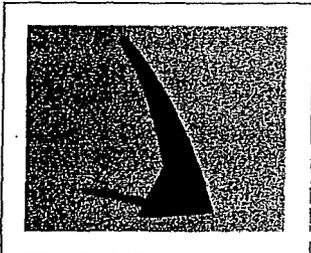
To use Cone-Fire Tuning, **IdLE** must display. Press **ENTER**, then 1. **ConE** will appear. Press **999**, **ENTER**. **rSLt** (for result) will display alternating with the current Tuning number. Enter the new number, then press **ENTER**. **ConE** will appear. Continue entering the values for the Cone-Fire program in the usual way. Once you change the Tuning number, Cone-Fire will remain adjusted to that number until you change it again.

The large cone on the kiln shelf should be visible through a peephole. Avoid exposure to cool air by keeping the cone at least 3" away from the peephole. Program the DTC 800C for the cone on the shelf and fire. After cooling, check the cone:

The cone bent to 6 o'clock: In this case, the controller is matched to your kiln.

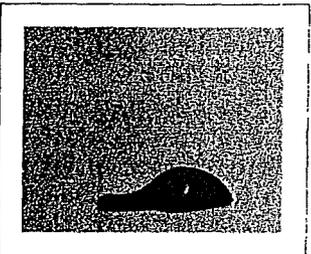


The cone did not bend far enough: Use a lower Tuning number for the next firing.



The cone bent too far: Use a higher Tuning number for the next firing.

Do not be overly concerned with achieving an exact 6 o'clock bend. The difference between a 3 o'clock and a 6 o'clock bend is only a few degrees. Cones, even from the same box, also vary slightly.



Ramp-Hold Mode

Ramp-Hold is available on both the DTC 800C and DTC 800.

In Ramp-Hold, the kiln fires in segments, or stages. Each segment has a firing rate, firing temperature, and hold (or soak) time. Ramp-Hold includes eight segments. Use only the number of segments you need per firing, from one to eight.

Rate is degrees of temperature change per hour, from 1° to 9999°F. (e.g., to increase temperature by 500° per hour, enter a rate of 500).

To control cooling, set the segment to a lower temperature than that of the preceding segment.

Storing User Programs in Ramp-Hold

A user program is a set of firing instructions to run the kiln in Ramp-Hold. A user program includes number of segments needed; firing rate, temperature, and hold (if any) for each segment; and an alarm temperature (if any).

Ramp-Hold Programming

As the program prompts you for segments, rate, temperature, etc., you will see values from the last firing. To use these again, just touch **ENTER**.

To fire without the Delay feature: Follow steps 1 through 9. Then press **START** twice.

- 1 Turn controller safety switch ON.
- 2 If controller displays **ErrP** or a flashing temperature, touch **ENTER**. **IdLE** will appear.
- 3 Touch **ENTER** then 4. **USER** will appear. Enter a number from 1 to 6 for the stored program desired.
- 4 Touch **ENTER**. **SEGS** will appear. Enter number of segments you will use. *ENTER 1.*
- 5 Touch **ENTER**. **RA 1** will appear. Enter firing rate for segment 1 (temperature change per hour; any temperature from 1° to 9999° F.). *ENTER 400°C.*
- 6 Touch **ENTER**. **°F 1** will appear. Enter the temperature you will be firing to in segment 1. *ENTER 250°C.*
- 7 Touch **ENTER**. **HLd1** will appear. Enter segment 1 hold (soak) time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). *ENTER 00.30*
- 8 Touch **ENTER**. Continue entering values for all segments.
- 9 Touch **ENTER**. **AlAr** will appear. Enter alarm temperature. (*Enter 9999* to turn alarm off.) Then touch **ENTER**.

IdLE will appear.

10 To set Delay Fire, touch **ENTER** then 3. **DELA** will appear. Enter delay time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). Then touch **ENTER**. (Delay zeroes out after each completed firing.)

→ *NO DELAY*
11 To start program, touch **ENTER** twice. **-On-** will appear, then kiln temperature. If a delay was programmed, **-ON-** will appear, then time remaining until start.

To stop the program during the firing cycle, touch **STOP** or turn the safety switch OFF. When program fires to completion, **CPLt** will appear alternating with total firing time in hours and minutes. Turn safety switch OFF when **CPLt** appears. To shut off the alarm when it sounds during a firing, press **ENTER**. *When Temp decreases AFTER the 20 minutes @ 250°C, HIT STOP.*

LET COOL DOWN ON ITS OWN.

Ramp-Hold can store up to six user programs even when the kiln is unplugged. Potters may want to store a program for crystalline glaze, another for a favorite red glaze, etc. Glass artists might want to store several programs for fusing their favorite types of glass and another for slumping.

When you enter Ramp-Hold mode, the first prompt to appear is **USER**. The **USER** prompt means, "Select a stored user program for this firing". If you are using Ramp-Hold for the first time, press 1. Then enter num-

ADDENDUM TO COMPLIANCE AGREEMENT WITH MITKEM CORPORATION

DECEMBER 18, 1976

CONTINUED

7. "USDA" soil samples may not be shipped to other facilities unless such facility has a valid permit and compliance agreement for imported soil or a valid compliance agreement for domestic soil. (See Item No. 3 for a list of such facilities)
8. "USDA" soil samples may not be used as a growing medium for plants and for the isolation and/or culture of organisms imported with the soil.
9. It is further agreed that the PPQ Officer may conduct an inspection of the laboratory facility and records concerning soil shipments at any time during normal working hours. All reasonable recommendations of the PPQ Officer to eliminate the spread of plant pests will be followed. Failure to comply with any of the stipulations of this Compliance Agreement may result in its revocation and the facility's authority to handle foreign and domestic regulated soil.

ALL SOIL RESIDUES, THEIR STORAGE CONTAINERS, FILTERS, ETC. SHALL BE TREATED WITH ONE OF THE FOLLOWING SCHEDULES:

Used soil storage containers may be treated by incineration.

DRY HEAT: Soil to be spread in layers 0.5 inches in depth to ensure uniform heat penetration.

<u>TEMPERATURE</u>		<u>EXPOSURE PERIOD</u>
110 - 120.5 C	(230 - 249 F)	16 HOURS *
121 - 154 C	(250 - 309 F)	2 HOURS *
154.5 - 192.5 C	(310 - 379 F)	30 MINUTES *
193 - 220 C	(380 - 429 F)	4 MINUTES *
221 - 232 C	(430 - 450 F)	2 MINUTES *

* Do not start counting time until the mass reaches the required temperature.

STEAM HEAT: 250 F at 15 lbs. pressure for 30 minutes.

Preheat laboratory autoclaves. Do not start counting time until pressure reaches 15 lbs.. Restrict soil depth to 2 inches when treating quantities of soil in trays. Restrict each package weight to 5 pounds or less when treating individual packages. Load with adequate spacing. Large commercial steam facilities which operate at pressures up to 60 lbs. psi will permit treatment of greater soil depth.

For large quantities of soil - use 250°F for 2 hours

**MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc**

STANDARD OPERATING PROCEDURE

For

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D

Rev. 10

Signature

Date

QA Director:

Chambers Lavelle

5/20/09

Lab Director:

Yuhai Tang

5/21/09

**MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc**

STANDARD OPERATING PROCEDURE

For

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8270D

Rev. 10

1. Scope and Application

This SOP describes the analysis of semivolatile organic compounds in solvent extracts of aqueous and soil samples using gas chromatography/mass spectrometry (GC/MS). The SOP covers the analyses according to protocols discussed in USEPA SW-846 Update IV, Method 8270D.

This SOP meets all of the requirements specified in the method. Where applicable, additional requirements for the US Department of Defense are also included.

To further familiarize with the procedures, the analyst is encouraged to consult the following instrument manuals and SOP:

- Department of Defense Quality Systems Manual for Environmental Laboratories: Version 3, January 2006 and 4.1, April 2009.
- Hewlett Packard HP 5972A MSD Hardware Manual.
- Mitkem Organic Prep Lab SOPs for Semivolatile sample extraction.

All sample matrices must be extracted and concentrated prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** or Peer analysts review the logbooks and data generated from this procedure and approve all reported results. The **Laboratory Director** or a member of senior management evaluates all laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

- 3.1 The samples are extracted using appropriate sample extraction methods (see separate SOPs for sample extraction) and, if necessary, sample clean-up procedures.
- 3.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer connected to the gas chromatograph.
- 3.3 Analytes eluted from the capillary column are introduced into the Mass Spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron-impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum of a five-point calibration curve.
- 3.4 A list of acronyms used in this SOP is included in **Table 1**.

The list of compounds to be analyzed and reported may vary from project to project. The SW-846 method contains several different lists of analytes, so there is no “official” EPA list of Method 8270D compounds. Mitkem typically analyzes samples for and reports a fairly extensive list of target analytes. Certain projects may also have additional compounds not on the normal Mitkem list. Alternatively, certain projects may have a shorter list of compounds than the normal Mitkem list. These project-specific lists of analytes are specified by the client through discussion with the Mitkem Project Manager who discusses the list with the Laboratory Supervisor. The lists are handled in the laboratory by the use of “sublists” in the Target data reduction and reporting software. In addition, when utilizing the Omega LIMS system, the sublist can be viewed using the SEL list option. SEL refers to the select list of target analytes requested by the client. It is used when this list differs from the “routine” analyte list. The list of Method 8270D compounds routinely analyzed by this method is presented in **Table 2**. Refer to the LIMS Test Information category/Test option/ limits of the testcode, for the most current MDL values. Those listed in **Table 2** may not be the most up to date.

- 3.4.1 Several options exist for the reporting extra compounds that are not on the routine Mitkem list. The ideal approach includes purchasing a primary calibration standard and a second source check standard. This is followed by the determination of method detection limits and inclusion of the extra compounds to the initial calibration standards as well as calibration verification standards and laboratory control spiking standards. Depending on the clients’ needs, alternate approaches may be appropriate. This may include single point calibration or searching for the compound as a Tentatively Identified Compound using Target software’s library search routines. The approach taken must be discussed with the

client prior to analyses, and if needed, sufficient documentation is included in the analysis report to enable validating the data. The analyst will be instructed by the lab supervisor as to what documentation is needed and what is required to be sent to the data reporting area for inclusion in the final report.

- 3.4.2 The Quality Control requirements contained in this SOP apply to the specific list of analytes being reported. QC criteria are to be evaluated for all project target analytes. While QC issues with non-routine analytes should be investigated, they are not critical if the compound is not reported. Mitkem's calibration standards and LCS/MS spiking solutions may contain additional compounds that are not reported for a particular project.

4. Sample Preservation, Containers, Handling and Storage

- 4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers. For semivolatile organic compound analysis by Method 8270D, water samples are collected in 1-liter amber glass bottles. Solid samples are collected in 8-ounce amber glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.
- 4.2 Sample extracts are transferred to the semivolatile organic analysis lab with appropriate sample preparation information. Extracts are stored at $4^{\circ} \pm 2^{\circ}\text{C}$, protected from light, in sealed crimp cap vials equipped with unpierced PTFE-lined septa and stored in a separate location from the analytical standards.
- 4.3 Extract hold-time for semivolatile organic compound analysis by Method 8270D is 40 days from date of sample extraction. The sample preparation holding times are covered in the corresponding extraction procedures SOPs.

5. Interferences and Potential Problems

- 5.1 Evaluate the raw GC/MS data to verify that interferences were not introduced during the extraction and/or clean up of the samples.
- 5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections. Whenever a highly concentrated (compounds detected at 200x MRL) sample is encountered, the subsequent sample must be evaluated for possible contamination. Presence of similar analytes in the subsequent

sample(s) will require reanalysis of these samples to establish that the analytes were not the result of contamination.

- 5.3 Method 8270D is not appropriate for multi-component analytes (aroclor, toxaphene, chlordane, etc.). Refer instead to Methods 8081 and 8082.
- 5.4 The following compounds may require special treatment when being determined by this method:
 - 5.4.1 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the GC, chemical reaction in acetone solution, and photochemical decomposition.
 - 5.4.2 N-nitrosodimethylamine and pyridine are difficult to separate from the solvent under the chromatographic conditions described; the filament should be turned on early enough to detect these compounds.
 - 5.4.3 N-nitrosodiphenylamine decomposes in the GC inlet and cannot be separated from diphenylamine.
 - 5.4.4 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC injection port is not properly maintained, or if reactive samples are analyzed previously in the sequence.

6. Equipment and Apparatus

6.1 Equipment:

- 6.1.1 There are four GC/MS in the semivolatile organic analysis lab. They are designated S1, S2, S3 and S4. S1 and S2 systems have similar configurations. Instruments S1 and S2 are Hewlett Packard (HP) Model 5890 GC interfaced to HP Model 5972A MSD. S3 system is a HP 6890 GC interfaces to a HP Model 5973 MSD. S4 system is a HP Model 6890N GC interfaced to a 5973 MSD. HP EnviroQuant Software is used to handle data acquisition. Target software by ThruPut is used for data reduction. Report generation is either performed directly with Target software, or the processed Target data are electronically transferred to the LIMS for reporting.
 - 6.1.1.1 The HP GC is fitted with an electron pressure controller (EPC) to allow constant carrier gas flow during the temperature ramp.
 - 6.1.1.2 A 30m x 0.25mm id (0.5 μ m film thickness) DB-5MS

Fused-silica capillary column (J & W) is used for the analyses. Equivalent columns including HP5MS (Hewlett Packard) and RTX5 (Restek) have been used with similar performance.

6.1.1.3 A HP Model 7673A autosampler is used for sample injection.

6.1.1.4 Instrument operating conditions are as follows:

General Gas Chromatography Conditions

Carrier Gas	Helium (99.999%)
Column Flow	about 1 mL/minute
Injector Temperature	275 to 295 °C
Transfer Line Temperature	290°C
Injection Volume	1 µL

General Mass Spectrometry Conditions

Mass Range	35-500 AMU
Scan Speed	at least 1 scan per second
Ionization Mode	70 eV positive ion

GC/MS program for DFTPP tune analysis:

DFTPP

GC/MS Program for Calibration Standards, Blanks, LCS/LCSD, MS/MSD and sample analysis:

BNA

Please note that the above are *general* instrument conditions and may be modified to respond to specific project needs.

6.2 Maintenance - The semivolatile GC/MS's are maintained according to the manufacturer's recommendation. The lab analyst performs preventive maintenance as discussed below.

6.2.1 On a daily basis whenever analyses are to be performed, replace GC septum and clean the injection port and liner, as well as the gold seal. After prolonged use, or after an analytical sequence in which high concentrations of target compounds are detected, it is at the analysts' discretion to evaluate the condition of the injection liner and gold seal to determine if they require replacement. Also clip up to 6" of the column. All preventative/routine maintenance is recorded in the associated Instrument Run Log.

- 6.2.2 If needed, the analytical column may be replaced; this is usually indicated by tailing of the polar compounds such as pentachlorophenol/benzidine, and/or when initial and continuing calibration verifications repeatedly fail to meet method requirements (especially for polar acidic or basic compounds). This type of maintenance is recorded in the Instrument Maintenance Logbook. The Instrument Maintenance is located in the Omega LIMS system and can be accessed using the category Analytical and option Instruments. All analysts have access to this function in LIMS. If help is needed, ask the Lab Supervisor for assistance. Document the manufacturer name and lot # of the new column in the LIMS Instrument maintenance log. The certificate for the column may be given to the QA Director for filing or may be scanned.
- 6.2.3 If the system constantly drifts out of DFTPP tune and/or the initial method requirements, the ion source will need to be cleaned. This maintenance will need to be recorded in the Instrument Maintenance Logbook.
- 6.2.4. There are two filaments in the mass spectrometer. If both filaments are blown, the HP 5972A MSD will be vented to replace both filaments. Whenever the ion source is opened for maintenance, the analyst should make sure both filaments are replaced. This would allow uninterrupted operation even if one filament were blown. This maintenance will need to be recorded in the Instrument Maintenance Logbook.

NOTE: After major maintenance such as the scenarios described in **sections 6.2.2 through 6.2.4**, an Initial Calibration (ICAL) is analyzed. Document the date of the ICAL in the resolution field in the LIMS Maintenance Logbook.

- 6.2.5 The rough-pump oil will be replaced at least once a year. Check the oil level periodically and add oil if needed. Document this maintenance as above.
- 6.2.6 Once a year, all GC/MS systems may undergo extensive maintenance by a skilled technician. When this occurs, collect all associated paperwork and enter relevant information in the LIMS maintenance log. The paperwork can be brought to the data reporting area for .pdf inclusion on the server.
- 6.2.7 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the instrument vendor for Telephone-consultation and if needed, schedule on-site repair. This information should be documented as in **Section 6.2.6**. In addition,

the resolution field in the LIMS Maintenance Logbook should be filled in fully.

- 6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD hardware manual.
- 6.4 Glassware
 - 6.4.1 Hamilton syringes (10µls, 25µls, 100µls, 250µls, 500µls, 1000µls). The manufacturer certifies syringe accuracy to ±1%.
 - 6.4.2 Vials – 2ml glass with PTFE-lined screw cap or crimp-cap tops.
 - 6.4.3 Mini-inert vials with on/off valve.

7. Reagents and Standards

- 7.1 Organic solvents – J.T. Baker ultra analyzed grade methylene chloride for standard preparation. Solvents are available to the GC/MS lab in 1-gallon liter bottles or in a smaller volume from the Organic Preparation Lab bulk storage distribution line. Always verify that the lot/serial number of the solvent has been approved before use. Use the solvent tracking log, logbook number 50.0016, kept in the Prep lab, to verify the solvent.
- 7.2 The standards used for this SOP are discussed below. ***Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.***
- 7.3 The laboratory will at all times archive or have on order one complete set of unopened ampulated standards (to include internal standards, surrogate standards and target analyte standards).

All primary standards received from vendors are logged into the SVOA Primary Standard Logbook. The standards are labeled *SPymmddX*,

Where: SP = Semivolatile Primary Standard
 yymmdd = date the standard is received
 X = the order the standard is logged into the Logbook on that date, in increasing alphabetical order.

- 7.4. Tune Standard: the tuning standard contains DFTPP, 4,4'-DDT, pentachlorophenol and benzidine. It is purchased from Restek (Cat. No.31615) at 1000 ug/mL.
- 7.5. Internal Standard: the internal standard is obtained from Cambridge Isotope as neat compounds. A 2000ug/mL solution of all compounds (1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthalene-d10,

Phenanthrene-d10, Chrysene-d12, and Perylene-d12) is prepared and transferred into 1mL aliquoted vials marked at the meniscus.

7.6. Primary Calibration Standards:

- 8270 MEGA Mix (Restek, Cat. No. 31850) at 500-1000ug/mL
- Benzidine Mixture (Ultra Cat. No. EPA-1071) at 5000ug/mL
- Acid surrogate (Restek Cat. No. 31087) at 10,000ug/mL
- B/N surrogate (Restek Cat. No. 31086) at 5000ug/mL
- 8270 add-on compounds, neat: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine. (Sigma Aldrich Cat. Nos. B1334, C2204, B34656, A10701 and Supelco Cat. No. 49085).

7.7. Second Source Standard: the second source standards are prepared at 200 ppm using one or more of the following:

- TCL BNA LCS Spike 100ug/mL (NSI Cat. No. WL-408-25)
- Acid Surrogate Mix (Restek Cat. No. 31083) at 7500ug/mL and
- Base/Neutral Surrogate Mix (Restek Cat. No. 31082) at 5000ug/mL
- Benzaldehyde 2000ug/mL (Restek Cat. No. 33017)
- Atrazine 1000ug/mL (Restek Cat. No. 32208)
- Caprolactam 1000ug/mL (Restek Cat. No. 31833)
- Biphenyl, prepared from neat, (ChemService Cat. No.PS-2032)

7.8 The working tune standard at 50ug/mL is prepared by adding 50uLs of the stock standard to a final volume of 1mL(*) with methylene chloride

7.9 The internal standard stock is prepared in-house at 2000ug/mL. A vial is opened and then emptied into a mini-inert vial equipped with an on/off valve for daily use. 20 uL are added to each 1.0 mL extract or standard.

7.10 An 8270 add-on Intermediate standard is prepared by weighing 0.05g neat compounds into 10 mL methanol for a concentration of 5000µg/mL.

7.11 An intermediate calibration standard at a concentration of 200µg/mL is prepared by combining the following volumes of primary standards and diluting to 5000uL(*) using methylene chloride:

<u>8270 MEGA Mix</u>	<u>1000uL</u>
<u>Benzidine Mix</u>	<u>200uL</u>
<u>Acid surrogate</u>	<u>100uL</u>
<u>B/N surrogate</u>	<u>200uL</u>
<u>8270 Add-on Mix</u>	<u>200uL</u>

7.12 Multi-level working calibration standards are prepared from the intermediate standard as follows(*):

Volume Volume Volume

	<u>Intermediate Standard(uL)</u>	<u>Methylene Chloride(uL)</u>	<u>Internal Standard(uL)</u>
10 ug/mL	50	950	20
20 ug/mL	100	900	20
50 ug/mL	250	750	20
80 ug/mL	400	600	20
120 ug/mL	600	400	20
160 ug/mL	800	200	20

- 7.13 Second source standard: a working second source standard at 50ug/mL is prepared in the same manner as the midpoint of the Initial Calibration.

(*) NOTE: Volumes above can be adjusted to make larger or smaller final volume of the Standards.

All of the working standards are labeled SWyymmddX,

Where: S = semivolatile

W = working standard

yymmdd = date the working standard is prepared

X = the order that the working standard is prepared on that date, in increasing alphabetical order

The working standards are protected from light and stored in the freezer (F7) at less than -10°C to -20°C. The standards are stored away from sample extracts to minimize cross contamination. All vials containing working standards must be labeled according to the current version of SOP 80.0001 Standard Preparation, Equivalency and Traceability. Be sure the vial label is not worn or difficult to read. Any vial whose label becomes worn or difficult to read should be re-labeled.

Working standard expiration dates are 12 months after they are prepared. Unopened ampulated standards' expiration dates are based on manufacturer's expiration dates. If no manufacturer's expiration date is provided the ampulated standards may be retained unopened for up to two years. Once an ampulated standard is opened it may be retained for one year from the date it was opened.

All of the standard preparation information is recorded in the appropriate standard logbook located in the lab. Document all necessary information related to the preparation of the standard including solvent lot number, volumes, expiration dates and standard Ids.

8. Procedure

To ensure the appropriate analyst is performing the analysis, the analyst's initials should be entered in the Enviroquant acquisition software (do not use the default value). The analyst processing and reviewing data will initial the Run Logbook when processing data.

8.1 Extraction - The methods in SW-846 typically used for sample extraction are as follows:

- Method 3510 extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.
- Method 3520 extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in a continuous liquid-liquid extractor and extracted with methylene chloride for a minimum of 18 hours.
- Method 3540 extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate, placed in an extraction thimble or between plugs of glass wool, and extracted using 1:1 v/v methylene chloride/acetone in a Soxhlet extractor.
- Method 3570 extracts small volumes of waste, sediment and soil samples for water-insoluble and slightly water-soluble organics. The samples are extracted with acetone and then with methylene chloride or hexane in a VOA vial.
- Method 3550 extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing powder, then extracted by ultrasonic extraction using 1:1 v/v methylene chloride/acetone.

8.2 Tuning:

The tune standard is prepared at 50 µg/mL. The GC/MS must be tuned to meet decafluorotriphenylphosphine (DFTPP) criteria every 12 hours when standards, samples or QC are to be analyzed.

All of the analysis information is to be recorded in the Instrument Logbook.

8.2.1. Procedure for performing tune - Use the GC/MS conditions in **Section 6.1.1.4** to perform the tune analysis.

8.2.2. Acceptance criteria for tune - The mass spectrum of DFTPP must be acquired in the following manner; three scans (the peak apex

scan and the scans immediately preceding and following the apex) are acquired and averaged; if needed, background subtraction will be performed and must be accomplished using no more than 20 scans prior to the elution of DFTPP. It is important that the analyst does not selectively add or subtract scans to generate the tune. This is the standard approach used by Target software.

For SW846 projects, the tune can also be obtained using one of the following procedures (1) use one scan at the peak apex, (2) use the one scan either directly preceding or the following scan, (3) use the average across the entire peak

Any composite spectrum that is obtained manually (not using the Target software approach) is required to be documented in the Instrument Run log. The analyst is required to document which scans were used for averaging as well as the scan that was used for background subtraction.

A typical mass spectrum and mass spectral listing of the tune in listed in **Figure 2**.

The acceptance criteria are as follows:

Mass	Ion Abundance
51	10 - 80% of Base Peak
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	10 - 80% of Base Peak
197	< 2.0% of mass 198
198	Base peak, or > 50% of mass 442
199	5.0 - 9.0% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present, < 24% of mass 442
442	Base Peak , or > 50% of mass 198
443	15 - 24% of mass 442

Once the mass spectrometer passes the DFTPP tune, all subsequent standards, samples, and QC associated with the tune must be analyzed using identical mass spectrometer instrument conditions.

Experience working with the instruments has shown the following:

- High m/z 51, this is due to dirty ion source.
- Low to zero abundance of m/z 365, this is due to lack of instrument sensitivity.
- High m/z 68 and/or 70, this is due to high background.

- Incorrect ratio of m/z 199/198 and 441/442/443, this is due to incorrect instrument mass resolution and/or threshold settings.

- 8.3 % Breakdown –the breakdown analysis is performed to ensure inertness of the GC system such that labile compounds will not decompose or be adsorbed in the chromatographic system. The system inertness is evaluated by assessing the integrity of DDT that is present in the tune solution. The breakdown of DDT to DDE and DDD can not exceed 20%. The breakdown is evaluated as follows: the mass chromatograms for m/z 235/165 are plotted to detect the presence of DDD, the mass chromatograms for m/z 246/318 are plotted to detect the presence of DDE. If these breakdown products are determined to be present, the area count of the total ion chromatogram (not single ion chromatogram) for DDT, DDE and DDD are integrated. The % breakdown is calculated as follows:

$$\% \text{ Breakdown} = \frac{\text{Area count of DDE} + \text{DDE}}{\text{Area count of DDT}} \times 100$$

If the breakdown exceeds the QC limit of 20%, consider replacing the injector seal and/or injection liner. Repeat the tune and evaluate the % breakdown.

- 8.4. Column performance check: the column performance check is performed to ensure the GC column is amenable to the analysis of polar compounds including acids and bases. The column performance check is evaluated by the tailing of benzidine and pentachlorophenol. The maximum tailing factor for each compound is 2. See **Figure 2** for instruction on how to calculate the tailing factor. If tailing factor is above 2, corrective action includes clipping off an additional length of the column. Repeat the tune and evaluate for tailing.
- 8.5 Initial Calibration - Initial calibration is performed after the instrument passes the tune, % breakdown requirements and column performance check. Initial calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial calibration will also be performed if continuing calibration analyses do not meet QA/QC criteria.

Six calibration standard solutions are required for all target and surrogate compounds. Standard concentrations of 10, 20, 50, 80, 120, and 160 ng/μL are required for the surrogates and all but nine of the target compounds. Nine compounds including 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, pentachlorophenol and benzoic acid require calibration at 20, 50, 80, 120 and 160 ng/μL. The lowest standard concentration is typically correlated with the reporting limits for the target analytes (standard concentration at or below the reporting limit).

This is the level closest to the method detection limit (MDL). There may be occasional requests to report results to limits that are below the lowest initial standard concentration. These must be documented and discussed in the project narrative. Any request for non-routine calibration should be discussed with the laboratory Supervisor and Project Manager to insure the resulting data meets project and method requirements and the procedures used and the quality of the data are fully documented.

DoD– The ICAL range shall consist of a minimum of 5 contiguous calibration points for organics, for all analytes reported. The low-level standard must be less than or equal to the reporting limit.

Several state and government programs have specific QA/QC Requirements and Performance Standards for the Initial Calibration. Refer to the individual state/government documents for more details. In particular, Dept. of Defense requires the evaluation of SPCC/CCC compounds in both the ICAL and CCV. See Attachment 1 for criteria.

After the calibration standards are prepared as directed in **Section 7.12**, the laboratory performs a six level calibration. Please note that for all target analytes except those with poor chromatographic performance discussed above, the relative response factor from the 20ng/uL level is not included in averaging the RRF. For the nine poor chromatographic performers, the relative response factor from the 10ng/uL level is not included and the 20ng/uL are used as the lowest calibration level.

8.5.1 Calculation for Initial Calibration:

From the multi-level level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$\text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where: A_x = area of the characteristic ion for the target compound to be measured

A_{is} = area of the characteristic ion for the associated internal standard

C_{is} = concentration of the internal standard

C_x = concentration of the compound to be measured

Please refer to **Table 3** for the list of target analytes and their associated internal standard. Please note that this is the Table 5 of the SW-846 Published Method 8270D.

The mean relative response factor is determined by averaging the 6 level RRF values.

The % relative standard deviation (%RSD) of the RRF is also calculated using:

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

$$\text{Where: Standard Deviation} = \sqrt{\sum (X_i - X)^2 / (n-1)}$$

Where: X_i = each individual value used to calculate the mean

X = the mean of n values

n = the total number of values = 5

8.5.2 Initial calibration acceptance criteria for SW-846 is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of -50% to $+100\%$ of the mean area response of the internal standard in all of the calibration levels.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- The recommended minimum RF for common compounds are listed in **Table 4** (also Table 4 of Published SW-846 8270D).
- The RSD for all target analytes and/or surrogate compounds must be $< 20\%$. The Target software will flag any compound whose RSD is greater than 20% . If the RSD of any target analytes and/or surrogate compounds is less than 20% , then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments. Experience with instruments S1 and S2 suggested that when the RRF decreases with increasing standard concentration, increasing the emission current from 50 mA to 75 mA and/or adjusting the electron multiplier voltage to make sure the area counts at 160 ng level do not exceed 10 million *will improve* system linearity.
- Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the initial calibration is deemed acceptable if the following conditions are met (in order of preference):

- (1) The method allows for a maximum of 10% of the target analytes and/or surrogate compounds to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier RSDs have a maximum of 50%.
- (2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)

m = slope of the line

x = concentration of the calibration standard

b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$.

- (3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)

a and b = slope of the curve

x = concentration of the calibration standard

c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be ≥ 0.99 .

- 8.5.3 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 50ug/mL standard prepared in **Section 7.13**. The acceptance criteria are as follows:

For routine SW 8270 analyses, the calculated value of the analyte in the ICV must be 70 – 130% of the expected value (35 – 65 ng/uL).

- For DoD analyses, the calculated value of the analyte in the ICV should be 80-120% of the expected value (40-60 ng/uL), with no allowance for poor performing compounds.

If the above criteria are not met, the analyst has to evaluate the integrity of the primary and second source standards. First, reanalyze the ICV. Preparation and analysis of a new initial calibration may be required.

- 8.5.4 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action included preparing fresh standards, source cleaning, changing GC column or injection liners. Document the actions and resolution in the LIMS maintenance log.
- 8.5.5 Initial calibration acceptance criteria must be met before any sample, blanks or QC are to be analyzed. There may be circumstances where project-specific criteria allow the use of an initial calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 20% of the non-CCC analytes (calibration check compounds are acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-phenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol and 2,4,6-trichlorophenol) to have %RSD > 30 or $r < 0.99$. This situation is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.
- 8.5.6 Upon the successful completion of the initial calibration, select the mid-point calibration (50 ug/mL standard) and update the reference spectra in Target.

8.5.7 Upon the successful completion of the initial calibration, the raw data are arranged in increasing concentration levels together with DFTPP tune. Raw data include chromatograms and quantitation reports plus any documentation of manual integrations. Refer to SOP No. 110.0008 for details on the need for and documentation of manual integration. A copy of the initial calibration summary listing the RRF and %RSD of each target analytes is also included. These raw data are to be filed separately for the four instruments.

8.5.8 Initial calibration data must be archived in the company's organic analysis calibration (OCAL) database. The information in **Section 8.5.7** is brought to the Data Reporting area and left in the tray for filing OCAL data. The Data Reporting department will scan the calibration printouts into the optical filing database for long-term archiving. This may be done at anytime after the ICAL is deemed acceptable.

8.6 Calibration verification - Calibration verification standards containing all of the target and surrogate compounds at 50ng on-column injection is performed every time samples are to be analyzed to ensure that the GC/MS system continues to meet instrument sensitivity and linearity requirements. An example of a continuing calibration chromatogram and quantitation report is included in **Figure 3**.

8.6.1 Frequency of Continuing Calibration - The continuing calibration standard must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

8.6.2 Procedure for Performing Continuing Calibration Verification - The continuing calibration verification is performed at 50ng injection. Calculate the % difference between the continuing calibration RRF and those from the most recent initial calibration. The % difference is determined as follow:

$$\% \text{ Difference} = \frac{\text{RRF}_c - \text{RRF}_i}{\text{RRF}_i} \times 100$$

Where:

RRF_c = relative response factor from continuing calibration

RRF_i = mean relative response factor from the most recent initial calibration which meets acceptance criteria

Use % drift when using least-squares calibration.

$$\% \text{ Drift} = \frac{\text{Conc}_c - \text{Conc}_t}{\text{Conc}_t} \times 100$$

$Conc_t$

Where: $Conc_c$ = concentration obtained from continuing calibration
 $Conc_t$ = theoretical concentration of standard

8.6.3 Continuing calibration acceptance criteria:

- The recommended minimum RF for common compounds are listed in **Table 4**.
- The % D must be $\leq 20\%$ (use % drift if using a regression fit model). Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the continuing calibration is deemed acceptable if the following condition is met:
- A maximum of 20% of the target analytes and/or surrogate compounds to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier %Ds have a maximum of 50%.
- No quantitation ion may saturate the detector.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (50 ug/mL) of the associated initial calibration.
- The internal standard area counts must be within +100% to -50% from that of the mid-point calibration (50 ug/mL) of the associated initial calibration.

Several states have specific QA/QC Requirements and Performance Standards for the Continuing Calibration. Refer to the individual state documents for more details.

8.6.4 Corrective Action for Continuing Calibration - Depending on which compound(s) fail(s) the criteria, corrective action includes preparing fresh standards, source cleaning, changing GC column or injection liners. Repeated failure to pass continuing calibration may necessitate performing new initial calibration. See **Attachments 1-3** for specific QC criteria and corrective action.

8.6.5 Continuing calibration acceptance criteria must be met before any samples or QC are to be analyzed. There may be circumstances where project-specific criteria allow the use of a continuing calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 10% of the non-CCC analytes (calibration check compounds are acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-phenylamine,

di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol and 2,4,6-trichlorophenol) to have %D > 30. This is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.7 Sample Analysis:

Prior to sample analysis, the sample extract and the internal standard prepared in **Section 7.9** are allowed to warm to room temperature to ensure complete dissolution of the high molecular weight internal standards.

Twenty microliters of the 2000 ppm internal standard solution is added to each of the sample extracts at 1mL final volume to ensure 40ng on-column amount. The internal standard volume will be adjusted for smaller extract volume. A 1uL aliquot of the extract is injected onto the GC/MS via an autosampler.

Target compounds identified in the sample extracts at concentration above the calibration range will be reanalyzed at dilution. These samples will be labeled with the DL suffix. For these re-analyses, an aliquot of the original extract will be diluted with methylene chloride. Additional aliquots of internal standards are then added to ensure an on-column injection of 40 ng of the internal standards. For the dilution analysis, the target compounds that exceeded the calibration range in the initial analysis should be detected above the mid-level calibration level (80 ng on-column).

- 8.7.1 Criteria for reporting dilution: the final dilution analysis is always reportable. This analysis should have the concentration of the most concentrated compound near or above the mid-level point of the calibration range.
- 8.7.2 Normally, the initial analysis result will be reported as long as QC criteria are met.
- 8.7.3 If the laboratory has prior information that a sample may contain concentrations of target and/or non-target compounds exceeding the instrument calibration range, or if the extracts are dark and viscous, the initial analysis may be performed at dilution. The initial analysis at dilution is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.7.4 If the initial and dilution analysis together demonstrate matrix interference, such as surrogate/internal standard area counts out of the QC limit in both analyses, both sets of data are typically reported. Also, if the initial analysis provides important information to the project, it should be reported, with the QC exceedences noted on the data sheets (eg: flagged surrogate recovery in Form 2, flagged internal standard area counts in Form 8 and "E" data qualifier in Form 1). These will be incorporated and discussed in the project narrative.

8.8. Analytical Sequence: The following sequence is recommended:

Initial Batch

1. Tune
2. ICal Standard #1
3. ICal Standard #2
4. ICal Standard #3
5. ICal Standard #4
6. ICal Standard #5
7. ICal Standard #6
8. ICV (second source)
9. Method Blank
10. LCS
- 11-18. Samples (< 8)
19. Tune (as required per 12 hr.)
20. CCV (as required per 12 hr.)
21. Method Blank
22. MS
23. MSD
- 24-31. Samples (< 8)

Middle/Final Batch

1. Tune
2. CCV
3. Method Blank
4. LCS
- 5-12. Samples (< 8)
13. CCV
14. Method Blank
15. MS
16. MSD
- 17-24. Samples (< 8)

In instances where there is no Method Blank, LCS, MS/MSD with the batch of samples to be analyzed, analyze an instrument blank after the successful completion of the calibration verification. Analyze colorless or light colored extracts first and arrange the dirty sample extracts towards the end of the analytical sequence.

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative Retention Time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$\text{RRT} = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Comparability of mass spectra - The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative abundance greater than 30% must be present in the sample spectrum
- the relative intensities of ions specified above must agree within $\pm 30\%$ between the standard and sample spectra
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered; this may be due to potential co-eluting interferences
- if the criteria above are not met but in the technical judgement of the analyst that the identification is correct, the lab will report the identification and proceed with the quantitation

9.2 Identification of non-target compounds [tentatively identified compounds (TICs)] - Client may request the analysis of TICs. Non-target compounds will be searched using the NIST/EPA/NIH library. The non-target compound will be reported as part of the analysis requirement if:

9.2.1 The client requires a full data package deliverable, including Mitkem level 4 or New York ASP-B reporting format (exceptions are projects that have a short list of target analytes such as TCLP, PAH, acid compounds only, base-neutral compounds only, STAR list or projects that the client specified no TIC reporting).

9.2.2 The non-target compounds will be identified and reported if:

- its response is greater than 10% of the closest eluting interference free internal standard
- its retention time is within the range of 30 seconds before the elution of the first target compounds (early eluting aldol condensation products are thus not reported), and 3 minutes after the elution of the last target compound
- Unless specified, up to **20** TIC are to be reported

9.2.3. Guidelines for making tentative identification :

- Ions greater than 10% in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$.

- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in sample spectrum but not in the reference spectrum should be reviewed for co-eluting interferences.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed with caution because of background contamination and/or co-eluting interferences.
- The lab shall report pesticide target compounds as semivolatile TIC.
- The lab shall not report volatile target compounds.

The non-target compounds will be reported as “unknown” if no valid tentative identification can be made (as based on analysts’ interpretation). If possible, try to give classification of the unknown, eg. unknown aliphatic hydrocarbon, unknown halogenated etc.

If the Quality (Qual) of the match as determined by the library search program is above 85%, it typically meets the criteria above, and is considered a tentative identification. If the Qual is less than 85%, the match typically does not meet the criteria above, and is usually identified as “unknown”.

- 9.3 Determining the Concentration of Target Compounds - Sample data are reported in units of $\mu\text{g/L}$ for aqueous samples and ug/Kg dry weight basis for solid samples. For aqueous samples, results are reported to one significant figure if the value is $\leq 10 \text{ ug/L}$. At greater concentration, the results are reported to two significant figures. Solid sample results will be reported in dry weight unless otherwise specified for unusual sample matrices. Results for solid sample are reported to two significant figures.
- 9.4 Rounding Rule – Analysis results are to be rounded according to the current EPA guidelines.
- 9.5 The average RRF from the initial calibration is used to quantitate the target compounds. It is important to note that the concentrations of the target compounds do not exceed the calibration range. Any target analyte that exceeds the calibration range will be diluted and re-analyzed as discussed in **Section 8.7**.
- 9.5.1 Manual integration will be performed if needed and documented according to the current revision of SOP No. 110.0008, Manual Integration of GC and GC/MS Chromatographs. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baselines and resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce/increase surrogate peak

area to be within recovery limits. The use of manual integration to purposefully modify non-compliant data is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. Hardcopies of the before and after ion chromatograms of the quantitation ions will be generated. The analyst will further initial and date the manual integration on the quantitation report with the proper reason code per SOP No. 110.0008.

Target compounds identified are quantitated using the following equations:

$$\text{For aqueous samples, Concentration } \mu\text{g/L} = \frac{(A_x) (I_s) (V_t) (Df)}{(A_{is}) (RRF) (V_o) (V_i)}$$

$$\text{For solid samples, Concentration } \mu\text{g/Kg} = \frac{(A_x) (I_s) (V_t) (Df)}{(A_{is}) (RRF) (W_s) (V_i) (S)}$$

Where: A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion of the associated internal standard

I_s = amount of internal standard added in nanogram

RRF = relative response factor

V_o = volume of water extracted in milliliters = 1,000

V_t = volume of the sample extract in milliliters = 1

Df = dilution factor

W_s = weight of soil extracted in grams

S = % solid

- 9.5 Determining the concentration of non-target compounds - An estimated concentration for non-target compounds is determined using the closest eluting internal standard free of interference. The formula to calculate the concentration is the same as those for water and soil samples described above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the associated internal standard. A RRF of one (1) is assumed. An estimated concentration must be calculated for all tentatively identified compounds as well as these identified as unknown.
- 9.6 Acceptance Criteria for Sample Analysis - Acceptance criteria are as follows:
- The sample must meet both extraction and analysis holding time.

- The sample must have a compliant tune, initial calibration and continuing calibration.
- The sample must have a compliant method blank.
- The sample must have a compliant LCS and/or LCSD
- The surrogate recovery per this SOP (**Section 10.6**) or client-specified criteria must be met.
- All of the target analyte concentrations should be within the calibration range.
- Excluding the solvent front or the aldol condensation peak for solid extract analysis, no ion should saturate the detector.

In addition,

- Area count of each of the internal standards in the inclusive range of - 50% to + 100% of the response of the continuing calibration.
- Retention time of each of the internal standards must not shift more than \pm 0.5 minute from the continuing calibration.

When the above two criteria are not met, the laboratory will re-analyze the sample at the same concentration or at dilution, unless similar results were found for its associated MS/MSD. Dark and viscous extracts with an unresolved complex mixture consisting of hydrocarbons usually results in the depression of the last two eluting internal standards and subsequent high recovery of terphenyl-d14. In these instances, the samples may not require re-analysis. The supervisor or project manager should be consulted for further guidance.

See **Attachments 1-3** for specific guidelines. Please note that these two criteria must be met for interference-free QC samples including method blank and LCS/LCSD.

- 9.7 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

$$\% \text{ Recovery } (\%R) = 100 \times (\text{SSR}-\text{SR})/(\text{SA})$$

Where: SSR = spiked sample result
 SR = sample concentration
 SA = spike added

- 9.8 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$\text{RPD} = \frac{(\text{D1}-\text{D2})}{(\text{D1}+\text{D2})/2} \times 100$$

Where: RPD = relative percent difference

D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

- 10.1 All standards made from a primary standard expire on or before the primary standard's expiration date. All standards must be labeled with the expiration date.
- 10.2 Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results. All analysts must have read this SOP and asked questions and received explanation for the areas they are not familiar with. This SOP should be referred to often, and used as a reference for this procedure. Details of the procedure for documenting analyst proficiency can be found in the current revision of SOP 80.0016.
- 10.3 Method blanks - A method blank is extracted with every batch not to exceed 20 samples (excluding LCS/LCSD, MS/MSD).

Acceptance criteria for the method blank are as follows:

- The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.
- The concentration of the target compounds in the method blank must be less than the Reporting Limit; the concentration for common laboratory contaminants such as phthalate esters, must not exceed the 5 times the Reporting Limit.

For DoD projects, the concentration of the target compounds in the method blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as phthalate esters, must not exceed the Method Reporting Limit.

Any sample associated with a blank that fails these criteria checks shall be re-extracted in a subsequent preparation batch, except when the sample analysis resulted in a non-detect for the compound that failed the criteria.

If no sample volume remains for re-extraction, the results shall be reported with appropriate data qualifying codes. The "B" qualifier is applied to positive sample results on Form 1 or LIMS Level 2 data sheet when the same compound is detected in the blank. For semivolatile analysis, the most common lab contaminant is bis (2-ethylhexyl) phthalate (BEHP).

- 10.4 Lab Control Sample (LCS) – A Lab Control Sample is a weight or volume of a clean reference matrix (sodium sulfate or DI water) that is spiked with

all target analytes and surrogates and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples. Where applicable, a Lab Control Sample Duplicate (LCS) will also be performed to evaluate reproducibility.

10.4.1 Acceptance criteria for LCS:

- General acceptance: compliant surrogate recovery
- For regular SW8270 projects, the recovery is evaluated against the established in-house limits. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits. See **Attachment 2** for *DoD* QC limits.
- If target analytes are outside of the acceptance limits, corrective action is required. Project-specific requirements, if available, will dictate the corrective action performed. See **Attachment 3** for further guidance.
- Due to the large number of target analytes, some recoveries may be out.

Per *DoD* requirements, analyses of <11 analytes, no marginal exceedences (ME) are allowed. For the analysis of 11-30 analytes (typical PAH analysis), one ME is allowed; for the analysis of 31-50 analytes, two ME are allowed; for 51-70 analytes, three ME are allowed; for the analysis of 71-90 analytes (typical routine 8270D analysis), four ME are allowed; for the analysis of >90 analytes, five ME are allowed. See **Attachments 1 and 2** for further guidance.

- Reporting LCS Results – If any compounds are outside of the acceptance limits, their recoveries are qualified with the “*” flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged with an “S” on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

- 10.5 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain elevated concentrations of target analytes, the laboratory should perform one matrix spike and a separate unspiked duplicate. For clean samples and those without documented history, matrix spike/matrix spike duplicate analyses are performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will routinely perform matrix spike and matrix spike duplicate.

10.5.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer the question posed above.

Control limits are the same as those discussed in **Section 10.4** (same as LCS aqueous and soil), but are used as advisory guidelines. This is especially true when the native sample exhibited a matrix effect or the spike concentrations are less than the native sample concentration. If the MS/MSD do not meet the in-house criteria, see **Attachment 3** for corrective action guidelines.

For *DoD* projects, the %RPD limits for the duplicate set is 30%. See **Attachment 1** for corrective action.

10.5.2. Reporting the Duplicate Matrix Spikes-If any compounds are outside of the acceptance limits, their recoveries and/or RPD are qualified with the “*” flag on the recovery MS/MSD summary report (Form 3) for CLP-type data reports, and flagged with an “S” on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks and LCS/LCSD, MS/MSD will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1. Acceptance criteria - The percent recovery of each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS must be within the in-house acceptance windows with the exception of one surrogate per fraction allowed out. Outliers should have at least 10% recovery with the exception of Phenol-d5 in an aqueous sample. This compound is an extremely poor performer.

- For *DoD* projects:

	<u>Solid</u>	<u>Aqueous</u>
• 2-Fluorophenol	35-105	20-110
• Phenol-d ₅	40-105	no limits
• 2,4,6-Tribromophenol	35-125	40-125
• Nitrobenzene-d ₅	35-100	40-110
• 2-Fluorobiphenyl	45-105	50-110

• p-Terphenyl-d₁₄ 30-125

50-135

10.6.2. Any **sample**, which fails to meet the above criteria, will be subjected to re-extraction. There may be instances when the recovery exceedance is matrix related. For example, samples with high concentration of non-target hydrocarbons will have depressed internal standard area counts and resulting “elevated” recovery of the associated surrogate compound such as terphenyl-d₁₄. In these instances where the recovery has a high bias due to sample matrix, the sample is not re-extracted. The sample extract is however re-analyzed to confirm the matrix effect. Once confirmed, both data sets are reported and the occurrence mentioned in the report narrative.

Any **method blank**, which fails to meet the above criteria, will trigger re-extraction of the entire preparation batch. In the event that the surrogate recoveries are above the upper QC limits and no target analytes are detected, the Technical Director or Laboratory Manager should be notified for guidance regarding re-extraction. In the event that re-extraction is warranted but no sample volume remains, notify the Lab Manager and Project Manager, report the data and note the issues in detail on the data review checklist for inclusion in the project narrative. See **Attachment 3** for corrective action guidelines.

10.6.2.1. If re-extraction and re-analysis of the sample demonstrate similar recovery performance, both sets of results will be reported to demonstrate matrix-related problems.

10.6.2.2. Re-extraction is not required for the sample, if the recovery is out of the QC limits for both the sample and its duplicate matrix spikes.

10.6.3. Reporting of Surrogate Recoveries-All surrogate outliers will be flagged with an “*” on the surrogate recovery report (Form 2) for CLP-type data reports, and flagged with an “S” on Level 2 LIMS type data reports.

10.7. Annually, MDL studies are conducted to establish the limit of detection applicable to this method. MDL verification at approximately 1-4 x MDL is analyzed after the study which also sets the DoD QSM Version 4.1 Limit of Detection (LOD). MDL verification must be analyzed quarterly on each instrument used for DoD program work. Please refer to the Mitkem SOP No. 80.0005 Determination of Method Detection Limits for more detail

11. Data Validation and Reporting

- 11.1 All raw data, including calibrations, QC results, and samples results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, sample and extract transfer logs, and instrument logs are reviewed and signed by the appropriate area supervisor. 100% of the data is reviewed. The QA Director randomly reviews 10% of the data reported by the laboratory.
- 11.2 Analysts transfer organic data report forms, data review checklist(s) and raw data to the reporting group for assembly into a final report. The data submitted for report preparation is dependent on project requirements and is subjected to further review by a data reviewer. The project manager reviews the data for reasonableness and writes the project narrative prior to releasing the report to the customer.

12. Corrective Action Procedures

- 12.1 Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. QC corrective action tables for *DoD* are presented in **Attachments 1 and 2**. An overview of the corrective actions and associated documentation is listed in **Attachment 3**.
- 12.2 Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedure SOP No. 80.0007. Starting in 2006, corrective actions are recorded in the Omega LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

13. Health and Safety

- 13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.
- 13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8270D, SW-846 Test Methods for Evaluating Solid Wastes, Revision 4, February 2007.
2. "Quality Systems Manual for Environmental Laboratories" Department of Defense, Final Version 3, January 2006 and Final Version 4.1, April 2009.

Attachments:

1. **Table 1:** List of Acronyms
2. **Table 2:** Semivolatile Analyte list and Method Reporting Limits (RLs).
3. **Table 3:** Internal Standard/Analyte list (Table 5 from Published Method).
4. **Table 4:** Suggested RRF per SW8270D(Table 4 from Published Method).
5. **Figure 1:** Calculation of Peak Tailing Factors.
6. **Figure 2:** DFTPP Tune and Chromatogram.
7. **Figure 3:** Continuing Calibration Standard Chromatogram and Quantitation Report.
8. **Attachment 1:** DoD Specific QC Requirements: DoD-B SW846 box, Table B-3 and F-4.
9. **Attachment 2:** DoD Specific QC Control Limits: Tables D-6, D-7, G-6 and G-7.
10. **Attachment 3:** Overview of Corrective Action and Documentation Examples.
11. **Attachment 4:** Additional QA/QC Requirements for MA-DEP.

Table 1
List of Acronyms

1,2-DCB	1,2-Dichlorobenzene
1,3-DCB	1,3-Dichlorobenzene
1,4-DCB	1,4-Dichlorobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
AFCEE	Air Force Center for Environmental Excellence— procedures used for selected projects—to be replaced by DOD QSM procedures.
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense (including Army, Navy, Air Force)
LCS	Lab control sample
MDL	Method detection limit
MQL	Method quantitation limit
ME	Marginal Exceedence
MS	Matrix spike
MSD	Matrix spike duplicate
QSM	Quality Systems Manual for DoD work
RL	Reporting Limit (occasionally referred to as PQL or Practical Quantitation Limit, or MRL or Method Reporting Limit)

Table 2

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W
 Test Number: SW8270A
 Test Name: SVOA by GC-MS
 Matrix: Aqueous

Units: µg/L

DefaultPrep: BNA_W_PR

Conversion: 1000.0000
 Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0001	0001	Phenol	0.74000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0002	0001	Bis(2-chloroethyl)ether	0.86000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0003	0001	2-Chlorophenol	0.71000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0004	0001	1,3-Dichlorobenzene	0.66000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0005	0001	1,4-Dichlorobenzene	0.63000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0006	0001	1,2-Dichlorobenzene	0.66000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0007	0001	2-Methylphenol	1.20000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0008	0001	2,2'-oxybis(1-Chloropropane)	0.99000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0009	0001	4-Methylphenol	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0010	0001	N-Nitroso-di-n-propylamine	0.84000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0011	0001	Hexachloroethane	0.73000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0011	0002	Nitrobenzene	0.90000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0012	0002	Isophorone	0.74000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0014	0002	2-Nitrophenol	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0015	0002	2,4-Dimethylphenol	3.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0016	0002	2,4-Dichlorophenol	0.98000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0017	0002	1,2,4-Trichlorobenzene	0.81000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0018	0002	Naphthalene	0.67000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0019	0002	4-Chloroaniline	0.57000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0020	0002	Bis(2-chloroethoxy)methane	0.75000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0021	0002	Hexachlorobutadiene	0.70000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0022	0002	4-Chloro-3-methylphenol	0.92000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0023	0002	2-Methylnaphthalene	0.83000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0024	0003	Hexachlorocyclopentadiene	0.85000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0025	0003	2,4,6-Trichlorophenol	0.91000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0026	0003	2,4,5-Trichlorophenol	0.76000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0027	0003	2-Chloronaphthalene	0.89000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0028	0003	2-Nitroaniline	0.72000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0029	0003	Dimethylphthalate	0.78000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0030	0003	Acenaphthylene	0.70000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0031	0003	2,6-Dinitrotoluene	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0032	0003	3-Nitroaniline	0.93000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0033	0003	Acenaphthene	0.83000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W

DefaultPrep: BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS

Conversion: 1000.0000

Matrix: Aqueous

Units: µg/L

Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
A	0034	0003	2,4-Dinitrophenol	3.60000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0035	0003	4-Nitrophenol	0.90000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0036	0003	Dibenzofuran	0.72000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0037	0003	2,4-Dinitrotoluene	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0038	0003	Diethylphthalate	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0039	0003	4-Chlorophenyl-phenylether	0.74000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0040	0003	Fluorene	0.84000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0041	0003	4-Nitroaniline	1.10000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0042	0004	4,6-Dinitro-2-methylphenol	1.20000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0043	0004	N-Nitrosodiphenylamine	0.85000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0044	0004	4-Bromophenyl-phenylether	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0045	0004	Hexachlorobenzene	0.96000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0046	0004	Pentachlorophenol	0.72000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0047	0004	Phenanthrene	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0048	0004	Anthracene	0.94000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0049	0004	Carbazole	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0050	0004	Di-n-butylphthalate	0.92000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0051	0004	Fluoranthene	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0052	0005	Pyrene	0.89000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0053	0005	Butylbenzylphthalate	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0054	0005	3,3'-Dichlorobenzidine	0.84000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0055	0005	Benzo(a)anthracene	0.93000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0056	0005	Chrysene	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0057	0005	Bis(2-ethylhexyl)phthalate	2.50000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0058	0006	Di-n-octylphthalate	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0059	0006	Benzo(b)fluoranthene	1.50000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0060	0006	Benzo(k)fluoranthene	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0061	0006	Benzo(a)pyrene	0.97000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0062	0006	Indeno(1,2,3-cd)pyrene	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0063	0006	Dibenzo(a,h)anthracene	1.10000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
A	0064	0006	Benzo(g,h,i)perylene	0.85000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
I	0001	0001	1,4-Dichlorobenzene-d4	0.10000	10.00000	160.00000	N/A	N/A		01/15/09
I	0002	0002	Naphthalene-d8	0.10000	10.00000	160.00000	N/A	N/A		01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W
 Test Number: SW8270A
 Test Name: SVOA by GC-MS
 Matrix: Aqueous

Units: µg/L

DefaultPrep: BNA_W_PR

Conversion: 1000.0000
 Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
I	0003	0003	Acenaphthene-d10	0.10000	10.00000	160.00000	N/A	N/A		01/15/09
I	0004	0004	Phenanthrene-d10	0.10000	10.00000	160.00000	N/A	N/A		01/15/09
I	0005	0005	Chrysene-d12	0.10000	10.00000	160.00000	N/A	N/A		01/15/09
I	0006	0006	Perylene-d12	0.10000	10.00000	160.00000	N/A	N/A		01/15/09
S	0001	0002	Nitrobenzene-d5	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
S	0002	0003	2-Fluorobiphenyl	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
S	0003	0005	Terphenyl-d14	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
S	0004	0001	Phenol-d5	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
S	0005	0001	2-Fluorophenol	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
S	0006	0004	2,4,6-Tribromophenol	0.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2-Nitrophenol-d4	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2-Picoline	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4-Aminobiphenyl	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	1,2,3,4-Tetrachlorobenzene	0.77000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	1,2,3,5-Tetrachlorobenzene	0.64000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	4-Chlorophenol	0.55000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4-Chloroaniline-d4	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	1,2,3-Trimethylbenzene	0.46000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	1,2,4,5-Tetrachlorobenzene	0.66000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0002	3-Chlorophenol	0.54000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	1,2-Dichlorobenzene-d4	0.83000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	3,3'-Dimethylbenzidine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4,6-Dinitro-2-methylphenol-d2	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	1,2-Diphenylhydrazine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	1,3,5-Trichlorobenzene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	3/4-Chlorophenol	0.55000	20.00000	160.00000	N/A	N/A	12/26/08	02/24/09
X	0999	0000	3-Methylcholanthrene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	3,5-Dichlorophenol	0.51000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	3,4-Dichlorophenol	0.98000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	1,2,4-Trimethylbenzene	0.44000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2-Acetylaminofluorene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	2,4-Dichlorobenzotrifluoride	0.70000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	2,4,5-Trichlorotoluene	0.65000	10.00000	160.00000	N/A	N/A		01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W

DefaultPrep: BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS

Conversion: 1000.0000

Matrix: Aqueous

Units: µg/L

Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0003	2,3-Dichlorophenol	0.42000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	2,3,6-Trichlorophenol	0.60000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2,3,5-Trimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0002	2,3,5-Trichlorophenol	0.64000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	2,3,4-Trichlorophenol	0.79000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	2,5-Dichlorobenzotrifluoride	0.47000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0005	Benzidine	1.70000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0003	2,3,4,6-Tetrachlorophenol	1.10000	25.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	1-Naphthylamine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	2,6-Dichlorophenol	0.50000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	1,4-Dioxane	3.50000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	1-Methylphenanthrene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0000	2-Naphthylamine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	2-Butoxyethanol	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	2-Chloroaniline	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	1-Methylnaphthalene	0.79000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	1-Chloronaphthalene	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	1-Chloro-2-nitro-4-(trifluoromethyl)benzene	0.46000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	2-Chlorophenol-d4	1.20000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	2-Ethyl-1-hexanol	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	1,4-Naphthoquinone	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	1,4-Naphthoquinoline,1-oxide	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2,4-Dichlorophenol-d3	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	1,4-Dioxane-d8	0.82000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	2,5-Dichlorophenol	0.65000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	1,3-Dinitrobenzene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	2,6-Dimethylnaphthalene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0000	Missouri - Oil Range Organics	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0004	Azobenzene	0.89000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	N-Nitrosopyrrolidine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	N-Nitrosopiperidine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	N-Nitrosomorpholine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	N-Nitrosomethylethylamine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W

DefaultPrep: BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS

Conversion: 1000.0000

Matrix: Aqueous

Units: µg/L

Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0001	N-Nitrosodimethylamine	0.93000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	N-Nitrosodiethylamine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	o-Toluic Acid	0.02000	20.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	N,N-Dimethylformamide	0.43000	10.00000	160.00000	N/A	N/A	04/15/08	01/15/09
X	0999	0000	o-Toluidine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Missouri - Diesel Range Organics	50.00000	50.00000	5000.00000	N/A	N/A		01/15/09
X	0999	0000	Methyl Parathion	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Methyl methane sulfonate	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Methapyrilene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	m-Toluic Acid	0.02000	20.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Kepone	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Isosafrole	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Isodrin	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	N-Nitroso-di-n-butylamine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Phenol-d6	0.00999	0.00999	160.00000	N/A	N/A		01/15/09
X	0999	0000	Thionazin	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	Tetraethyllead	0.10000	10.00000	250.00000	N/A	N/A		04/08/09
X	0999	0000	Sym-Trinitrobenzene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Safrole	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	Pyridine	0.86000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Pyrene-d10	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Pronamide	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	O,O,O-Triethylphosphorothioate	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Phorate	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Fluorene-d10	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Phenacetin	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Perylene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0004	Pentachloronitrobenzene	1.20000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0003	Pentachlorobenzene	0.89000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	p-Toluic Acid	0.02000	20.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	p-Phenylenediamine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	p-(Dimethylamino)azobenzene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0004	Octachlorocyclopentene	0.62000	10.00000	160.00000	N/A	N/A		01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W

DefaultPrep: BNA_W_PR

Test Number: SW8270A

Test Name: SVOA by GC-MS

Conversion: 1000.0000

Matrix: Aqueous

Units: µg/L

Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0002	Phthalic anhydride	1.20000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	alpha-Terpineol	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Total SVOC	10.00000	10.00000	0.00000	N/A	N/A		02/24/09
X	0999	0001	Benzaldehyde	1.20000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0003	1,1'-Biphenyl	0.81000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0004	Atrazine	1.00000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Aramite-2	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Aramite-1	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Aramite	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Hexachloropropene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	Aniline	0.73000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Benzo(e)pyrene-d12	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0001	Acetophenone	0.84000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Acenaphthylene-d8	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	a,a-Dimethylphenethylamine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	7,12-Dimethylbenz(a)anthracene	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	5-Nitro-o-toluidine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0003	4-tert-Octylphenol	0.63000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4-Nitroquinoline-1-oxide	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4-Nitrophenol-d4	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Anthracene-d10	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Dibenz(a,j)acridine	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	4-Methylphenol-d8	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Famphur	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Ethyl parathion	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Ethyl methane sulfonate	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Disulfoton	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Diphenylamine	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Dimethylphthalate-d6	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Dimethoate	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Benzo(a)pyrene-d12	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Dibenzothiophene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09
X	0999	0000	Benzo(e)pyrene	0.00999	0.00999	0.00000	N/A	N/A		01/15/09

METHOD DETECTION / REPORTING LIMITS

Test Code: SW8270_W
 Test Number: SW8270A
 Test Name: SVOA by GC-MS
 Matrix: Aqueous

Units: µg/L

DefaultPrep: BNA_W_PR

Conversion: 1000.0000
 Updated: 01/15/2009

AT	RO	IS	Analyte	MDL	PQL	UQL	PR	Conv Factor	MDL Date	Last Update
X	0999	0000	Diallate	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	Chlorobenzilate	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0002	Caprolactam	1.40000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Bis (2-chloroisopropyl) ether	0.01000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0000	bis (2-Chloroethyl) ether-d8	1.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	Benzyl alcohol	0.85000	10.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0002	Benzoic acid	4.30000	20.00000	160.00000	N/A	N/A	01/11/08	01/15/09
X	0999	0000	Hexachlorophene	10.00000	10.00000	160.00000	N/A	N/A		01/15/09
X	0999	0001	Diethylenetriamine	1.00000	10.00000	160.00000	N/A	N/A		01/15/09

Table 3
(Table 5 from Published Method)

TABLE 5

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES
ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl- phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d ₈ (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d ₆ (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

TABLE 5
(continued)

Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
4-Aminobiphenyl	Benzydine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethyl aminoazobenzene	7,12-Dimethylbenz(a)anthracene
Hexachlorobenzene	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Terphenyl-d ₁₄ (surr)	Indeno(1,2,3-cd) pyrene
Pentachlorophenol		3-Methylcholanthrene
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(surr) = surrogate

Table 4
(Table 4 from Published Method)

TABLE 4

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

TABLE 4
(continued)

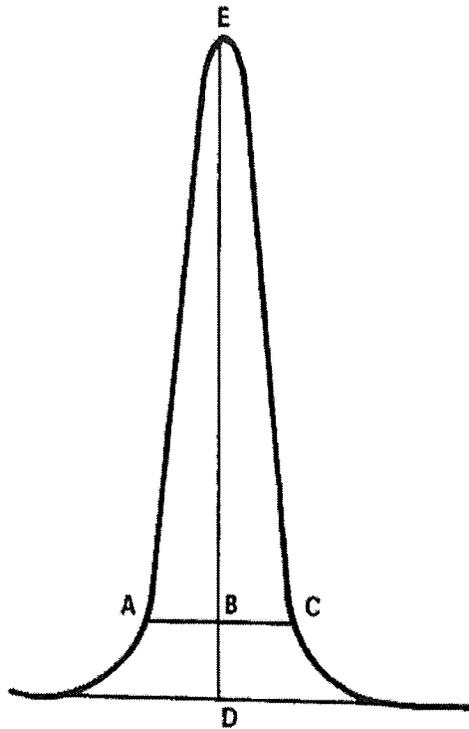
Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010

TABLE 4
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Figure 1

FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Figure 2

Date : 11-MAR-2009 16:05

Client ID: DFTPP3A

Instrument: S3.i

Sample Info: DFTPP3A,DFTPP3A

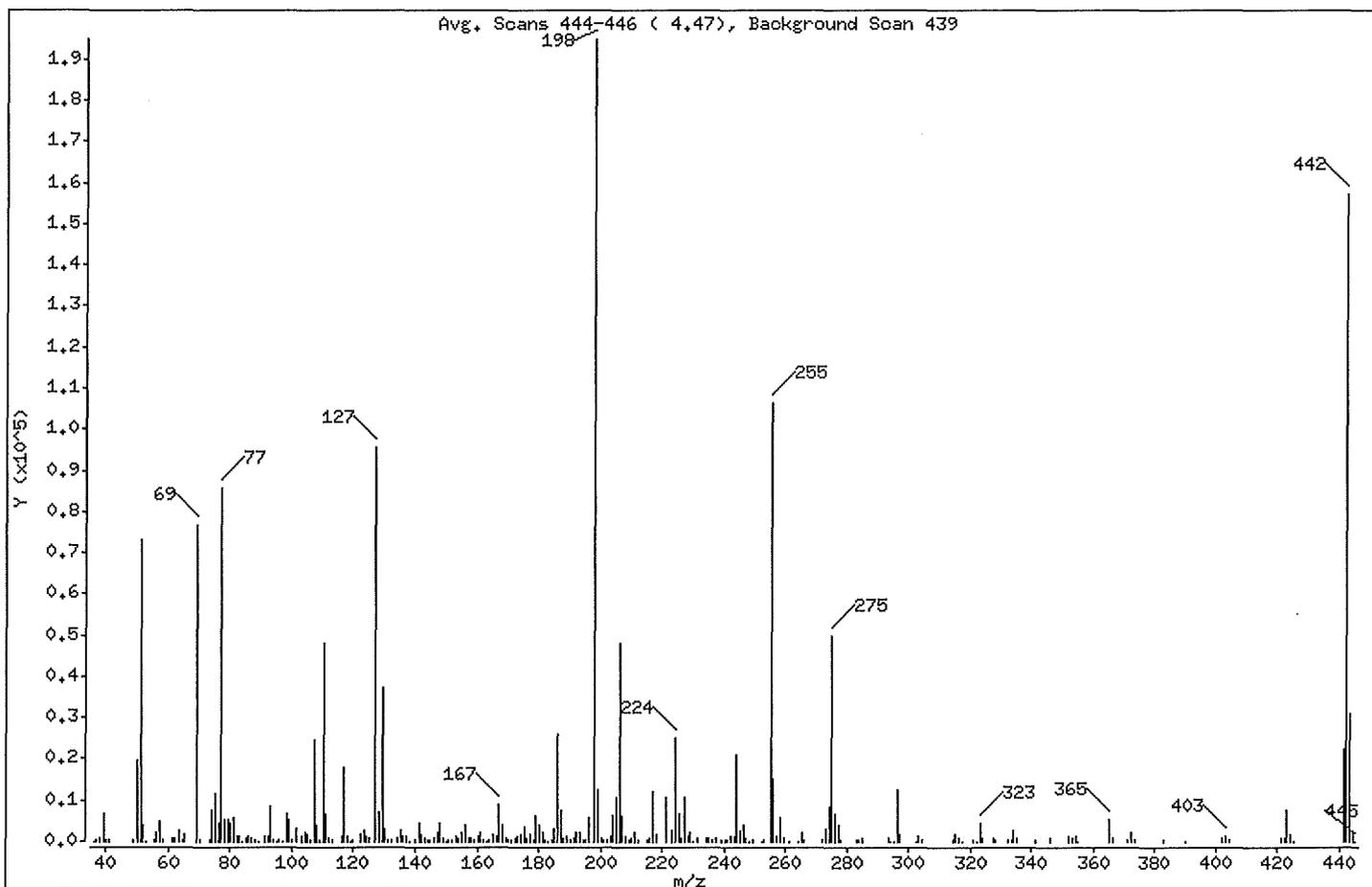
Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

1 dftpp



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	30.00 - 60.00% of mass 198	37.50
68	Less than 2.00% of mass 69	0.00 (0.00)
69	Mass 69 relative abundance	39.43
70	Less than 2.00% of mass 69	0.17 (0.44)
127	40.00 - 60.00% of mass 198	49.13
197	Less than 1.00% of mass 198	0.00
199	5.00 - 9.00% of mass 198	6.47
275	10.00 - 30.00% of mass 198	25.56
365	Greater than 1.00% of mass 198	2.87
441	Present, but less than mass 443	11.60
442	40.00 - 99.99% of mass 198	80.56
443	17.00 - 23.00% of mass 442	15.88 (19.71)

Date : 11-MAR-2009 16:05

Client ID: DFTPP3A

Instrument: S3.i

Sample Info: DFTPP3A,DFTPP3A

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

Data File: S3F6100.D

Spectrum: Avg. Scans 444-446 (4.47), Background Scan 439

Location of Maximum: 198.00

Number of points: 251

m/z	Y	m/z	Y	m/z	Y	m/z	Y
36.00	221	118.00	1410	185.00	3158	261.00	101
37.00	323	119.00	111	186.00	25840	264.00	134
38.00	904	120.00	321	187.00	7555	265.00	2191
39.00	6817	122.00	1731	188.00	746	266.00	421
40.00	548	123.00	2671	189.00	1539	272.00	234
41.00	296	124.00	1318	190.00	238	273.00	3177
49.00	292	125.00	899	191.00	767	274.00	8532
50.00	19760	127.00	95736	192.00	2204	275.00	49808
51.00	73072	128.00	7111	193.00	2181	276.00	6540
52.00	3947	129.00	37056	194.00	433	277.00	3937
53.00	104	130.00	3178	195.00	275	278.00	637
55.00	514	131.00	589	196.00	6044	283.00	408
56.00	2286	132.00	354	198.00	194880	284.00	270
57.00	4945	134.00	1060	199.00	12605	285.00	729
58.00	229	135.00	2906	200.00	1026	293.00	1080
61.00	810	136.00	1275	201.00	599	294.00	101
62.00	879	137.00	1450	202.00	368	295.00	121
63.00	2895	138.00	128	203.00	1161	296.00	12778
64.00	390	140.00	392	204.00	6456	297.00	1660
65.00	1586	141.00	4460	205.00	10780	302.00	222
69.00	76848	142.00	1701	206.00	48096	303.00	1534
70.00	336	143.00	952	207.00	6390	304.00	314
73.00	590	144.00	305	208.00	1509	314.00	596
74.00	7459	145.00	262	209.00	437	315.00	1594
75.00	11484	146.00	869	210.00	898	316.00	846
76.00	4359	147.00	2461	211.00	2040	317.00	101
77.00	85616	148.00	4708	212.00	295	321.00	409
78.00	5591	149.00	1058	215.00	502	322.00	105
79.00	5279	150.00	112	216.00	1005	323.00	4456
80.00	4426	151.00	540	217.00	12033	324.00	899
81.00	6030	152.00	436	218.00	1684	327.00	742
82.00	1387	153.00	1453	221.00	10983	328.00	473
83.00	1519	154.00	1021	223.00	2549	332.00	318
84.00	140	155.00	2449	224.00	25232	333.00	401
85.00	992	156.00	3867	225.00	6795	334.00	2711

Date : 11-MAR-2009 16:05

Client ID: DFTPP3A

Instrument: S3.i

Sample Info: DFTPP3A,DFTPP3A

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

Data File: S3F6100.D

Spectrum: Avg. Scans 444-446 (4.47), Background Scan 439

Location of Maximum: 198.00

Number of points: 251

m/z	Y	m/z	Y	m/z	Y	m/z	Y
86.00	1315	157.00	896	226.00	683	335.00	717
87.00	677	158.00	777	227.00	10710	341.00	451
88.00	309	159.00	552	228.00	1496	346.00	791
89.00	121	160.00	1418	229.00	2200	352.00	1474
91.00	1535	161.00	2197	230.00	139	353.00	855
92.00	1512	162.00	553	231.00	997	354.00	1452
93.00	8684	163.00	220	234.00	727	355.00	111
94.00	587	164.00	316	235.00	813	365.00	5589
95.00	119	165.00	1740	236.00	471	366.00	703
96.00	428	166.00	1427	237.00	815	371.00	284
97.00	116	167.00	9084	239.00	384	372.00	2338
98.00	6877	168.00	3968	240.00	300	373.00	578
99.00	5370	169.00	680	241.00	566	383.00	643
100.00	445	170.00	302	242.00	1362	390.00	108
101.00	3364	171.00	367	243.00	1454	402.00	872
103.00	1146	172.00	898	244.00	20976	403.00	1328
104.00	2046	173.00	1187	245.00	2840	404.00	366
105.00	1935	174.00	1992	246.00	4034	421.00	947
106.00	556	175.00	3541	247.00	888	422.00	954
107.00	24584	176.00	1025	248.00	104	423.00	7788
108.00	3904	177.00	1719	249.00	641	424.00	1633
109.00	367	178.00	599	252.00	126	425.00	108
110.00	48040	179.00	6315	253.00	420	441.00	22608
111.00	6956	180.00	4222	255.00	106568	442.00	156992
112.00	828	181.00	2174	256.00	15241	443.00	30944
113.00	372	182.00	351	257.00	1277	444.00	2780
116.00	1487	183.00	114	258.00	5790	445.00	131
117.00	17752	184.00	482	259.00	933		

Date : 11-MAR-2009 16:05

Client ID: DFTPP3A

Instrument: S3.i

Sample Info: DFTPP3A,DFTPP3A

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

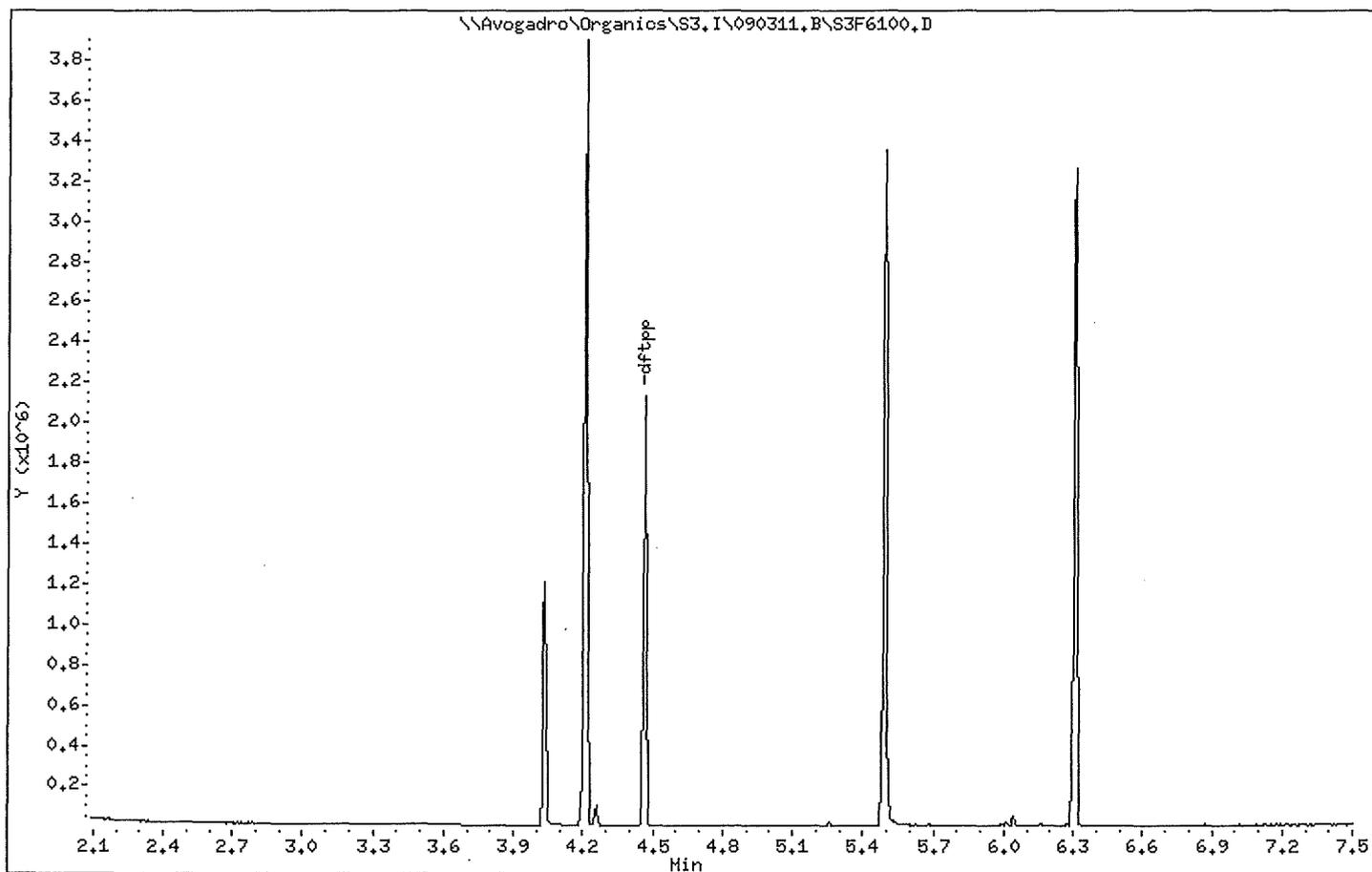


Figure 3

Data File: \\Avogadro\Organics\S3.I\090311.B\S3F6101.D

Date : 11-MAR-2009 16:22

Client ID: SSTD0503A

Sample Info: SSTD0503A,SSTD0503A

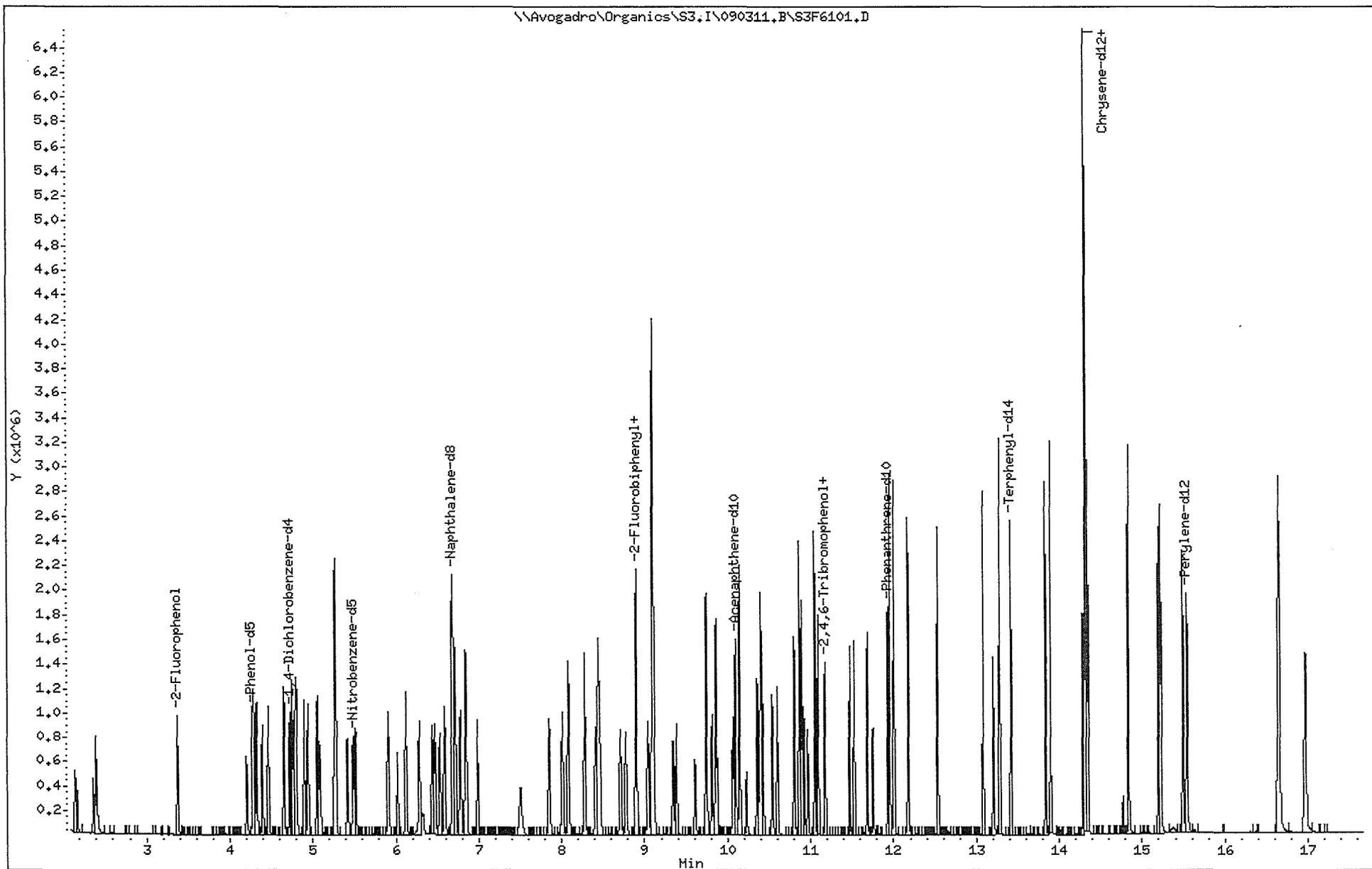
Volume Injected (uL): 1.0

Column phase: DB-5MS

Instrument: S3.i

Operator: CLM SRC: CLM

Column diameter: 0.25



Mitkem Laboratories

Data file : \\Avogadro\Organics\S3.I\090311.B\S3F6101.D
 Lab Smp Id: SST0503A Client Smp ID: SST0503A
 Inj Date : 11-MAR-2009 16:22
 Operator : CLM SRC: CLM Inst ID: S3.i
 Smp Info : SST0503A,SST0503A
 Misc Info : 2,3
 Comment :
 Method : \\Avogadro\Organics\S3.I\090311.B\s3_8270C_N.m
 Meth Date : 26-Mar-2009 14:07 S3.i Quant Type: ISTD
 Cal Date : 05-MAR-2009 19:14 Cal File: S3F5973.D
 Als bottle: 1 Continuing Calibration Sample
 Dil Factor: 1.00000
 Integrator: HP RTE Compound Sublist: mdlS3.sub
 Target Version: 4.14
 Processing Host: TARGET112

Concentration Formula: Amt * DF * Uf*(Vt/Vi)*(1/Vo) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	GPC Correction Factor
Vt	1000.000	Volume of final extract (uL)
Vi	1.000	Volume injected (uL)
Vo	1000.000	Volume of sample extracted (mL)
Cpnd Variable		Local Compound Variable

Compounds	QUANT	SIG	AMOUNTS					
			MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ng)
101 Benzaldehyde	77		4.205	4.205	(0.890)	138496	50.0000	76
6 Phenol	94		4.290	4.290	(0.908)	394810	50.0000	47
* 12 1,4-Dichlorobenzene-d4	152		4.723	4.723	(1.000)	182166	40.0000	
* 31 Naphthalene-d8	136		6.684	6.684	(1.000)	711711	40.0000	
114 1-Methylnaphthalene	142		8.281	8.281	(1.239)	636095	50.0000	49
39 2,4,6-Trichlorophenol	196		8.708	8.708	(0.862)	187168	50.0000	52
43 2-Nitroaniline	65		9.344	9.344	(0.925)	182244	50.0000	48
44 Dimethylphthalate	163		9.750	9.750	(0.966)	717938	50.0000	50
45 2,6-Dinitrotoluene	165		9.814	9.814	(0.972)	177359	50.0000	50
46 Acenaphthylene	152		9.862	9.862	(0.977)	987525	50.0000	50
* 48 Acenaphthene-d10	164		10.097	10.097	(1.000)	424778	40.0000	
49 Acenaphthene	153		10.145	10.145	(1.005)	615891	50.0000	49
52 Dibenzofuran	168		10.407	10.407	(1.031)	868730	50.0000	50
53 2,4-Dinitrotoluene	165		10.428	10.428	(1.033)	236226	50.0000	50
54 Diethylphthalate	149		10.802	10.802	(1.070)	721506	50.0000	49
55 Fluorene	166		10.872	10.872	(1.077)	747411	50.0000	50
56 4-Chlorophenyl-phenylether	204		10.899	10.899	(1.079)	336960	50.0000	50
59 N-Nitrosodiphenylamine	169		11.053	11.053	(0.926)	629385	50.0000	52
62 Hexachlorobenzene	284		11.529	11.529	(0.966)	188570	50.0000	51
* 64 Phenanthrene-d10	188		11.935	11.935	(1.000)	663482	40.0000	

Compounds	QUANT SIG				AMOUNTS		
	MASS	RT	EXP RT	REL RT	RESPONSE	CAL-AMT (ng)	ON-COL (ng)
65 Phenanthrene	178	11.956	11.956	(1.002)	1023449	50.0000	50
66 Anthracene	178	12.010	12.010	(1.006)	1048906	50.0000	51
67 Carbazole	167	12.181	12.181	(1.021)	1000945	50.0000	49
68 Di-n-butylphthalate	149	12.533	12.533	(1.050)	1193974	50.0000	50
71 Pyrene	202	13.286	13.286	(0.927)	1127502	50.0000	55
73 Butylbenzylphthalate	149	13.837	13.837	(0.966)	546178	50.0000	55
78 bis(2-Ethylhexyl)phthalate	149	14.318	14.318	(0.999)	743613	50.0000	53
* 76 Chrysene-d12	240	14.328	14.328	(1.000)	725485	40.0000	
82 Benzo(a)pyrene	252	15.498	15.498	(0.997)	1000579	50.0000	51
* 83 Perylene-d12	264	15.546	15.546	(1.000)	727298	40.0000	
84 Indeno(1,2,3-cd)pyrene	276	16.652	16.652	(1.071)	1106338	50.0000	50
85 Dibenzo(a,h)anthracene	278	16.657	16.657	(1.071)	927844	50.0000	50

SW
3/26/09

Attachment 1

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

**TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS,
PURPOSE, AND EVALUATION**

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18)	NA	Samples cannot be analyzed without a valid MDL.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270C only)	Daily prior to analysis of samples	Degradation \leq 20% for DDT	Correct problem then repeat breakdown check	Flagging criteria are not appropriate	No samples shall be run until degradation \leq 20%. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be observed.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	<p>1. <u>Average response factor (RF) for SPCCs:</u> VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs - ≥ 0.050.</p> <p>2. <u>RSD for RFs for CCCs:</u> VOCs and SVOCs - $\leq 30\%$ and one option below; Option 1: RSD for each analyte $\leq 15\%$ Option 2: linear least squares regression $r \geq 0.995$ Option 3: non-linear regression - coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)</p>	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within $\pm 25\%$ of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the initial calibration curve.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050 . 2. %Difference/Drift for CCCs: VOCs and SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. See section 5.5.10 and DoD clarification box 55.	Apply Q-flag if no sample material remains and analyte exceeds criteria.	
Internal standards verification	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within - 50% to + 100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch	No analytes detected $> \frac{1}{2}$ RL. For common laboratory contaminants, no analytes detected $> RL$.	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
LCS containing all analytes required to be reported, including surrogates	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MSD or sample duplicate	One per preparatory batch per matrix	RPD \leq 30% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS published by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification (See Box D-13)					
LOQ establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation \leq 20%.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<p>1. <u>Average response factor (RF) for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>SVOCs ≥ 0.050.</p> <p>2. <u>RSD for RFs for CCCs:</u> VOCs and SVOCs $\leq 30\%$ and one option below:</p> <p><u>Option 1:</u> RSD for each analyte $\leq 15\%$;</p> <p><u>Option 2:</u> linear least squares regression $r \geq 0.995$;</p> <p><u>Option 3:</u> non-linear regression-coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	<p><u>1. Average RF for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050.</p> <p><u>2. %Difference/Drift for all target compounds and surrogates:</u> VOCs and SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).</p>	<p>DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken.</p> <p>Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.</p>	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes detected $>$ $\frac{1}{2}$ RL and $>$ $\frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $>$ RL (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than \pm 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table F-4. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260 and 8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD \leq 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Attachment 2

**TABLE D-5. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846
METHOD 8260 SOLID MATRIX¹²**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane ¹³	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

**TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846
METHOD 8270 WATER MATRIX¹⁴**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120
Benzo[b]fluoranthene	81.8	12.1	45	120	35	130
Benzo[k]fluoranthene	84.6	13.2	45	125	30	135

¹⁴ A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

**Table G-5. LCS Control Limits for Volatile Organic Compounds SW-846 Method 8260
Solid Matrix³ (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Chloroform	98	9	70	125	65	135
Chloromethane	90	13	50	130	40	140
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane ⁴	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

**Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270
Water Matrix⁵**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
<u>Polynuclear Aromatics</u>						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120

⁵ A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX¹⁴

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benzo[g,h,i]perylene	80.5	14.1	40	125	25	135
Chrysene	82.1	8.9	55	110	45	120
Dibenz[a,h]anthracene	84.7	14.1	40	125	30	140
Fluoranthene	85.2	10.4	55	115	45	125
Fluorene	80.6	10.3	50	110	40	120
Indeno[1,2,3-cd]pyrene	84.3	13.6	45	125	30	140
Naphthalene	70.8	10.5	40	100	30	115
Phenanthrene	84.0	11.0	50	115	40	130
Pyrene	88.6	13.2	50	130	35	140
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	50	110	40	120
2,4,6-Trichlorophenol	80.7	10.7	50	115	40	125
2,4-Dichlorophenol	76.3	9.6	50	105	40	115
2,4-Dimethylphenol	68.8	13.5	30	110	15	125
2,4-Dinitrophenol	75.8	20.6	15	140	10	160
2-Chlorophenol	71.3	11.4	35	105	25	115
2-Methylphenol	73.3	11.7	40	110	25	120
2-Nitrophenol	75.8	12.4	40	115	25	125
3-Methylphenol/4-Methylphenol	71.3	13.0	30	110	20	125
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145
4-Chloro-3-methylphenol	78.6	10.7	45	110	35	120
Pentachlorophenol	77.6	13.3	40	115	25	130
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	20	110	10	125
4-Chloroaniline	62.2	15.6	15	110	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	40	125	30	140
Butyl benzyl phthalate	81.1	11.7	45	115	35	130
Di-n-butyl phthalate	84.8	10.3	55	115	45	125
Di-n-octyl phthalate	87.4	16.6	35	135	20	155
Diethyl phthalate	79.2	12.9	40	120	30	130
Dimethyl phthalate	75.9	16.9	25	125	10	145
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	35	130	20	145
N-Nitrosodimethylamine	67.9	14.1	25	110	10	125
N-Nitrosodiphenylamine	79.6	10.6	50	110	35	120
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	76.2	10.2	45	105	35	115
Bis(2-chloroethyl) ether	73.3	12.3	35	110	25	120
Bis(2-chloroisopropyl) ether	78.2	17.5	25	130	10	150
Hexachlorobutadiene	65.2	12.6	25	105	15	115
Hexachloroethane	60.9	11.1	30	95	15	105

**Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270
Water Matrix⁵ (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benzo[b]fluoranthene	81.8	12.1	45	120	35	130
Benzo[k]fluoranthene	84.6	13.2	45	125	30	135
Benzo[g,h,i]perylene	80.5	14.1	40	125	25	135
Chrysene	82.1	8.9	55	110	45	120
Dibenz[a,h]anthracene	84.7	14.1	40	125	30	140
Fluoranthene	85.2	10.4	55	115	45	125
Fluorene	80.6	10.3	50	110	40	120
Indeno[1,2,3-cd]pyrene	84.3	13.6	45	125	30	140
Naphthalene	70.8	10.5	40	100	30	115
Phenanthrene	84.0	11.0	50	115	40	130
Pyrene	88.6	13.2	50	130	35	140
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	50	110	40	120
2,4,6-Trichlorophenol	80.7	10.7	50	115	40	125
2,4-Dichlorophenol	76.3	9.6	50	105	40	115
2,4-Dimethylphenol	68.8	13.5	30	110	15	125
2,4-Dinitrophenol	75.8	20.6	15	140	10	160
2-Chlorophenol	71.3	11.4	35	105	25	115
2-Methylphenol	73.3	11.7	40	110	25	120
2-Nitrophenol	75.8	12.4	40	115	25	125
3-Methylphenol/4-Methylphenol	71.3	13.0	30	110	20	125
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145
4-Chloro-3-methylphenol	78.6	10.7	45	110	35	120
Pentachlorophenol	77.6	13.3	40	115	25	130
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	20	110	10	125
4-Chloroaniline	62.2	15.6	15	110	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	40	125	30	140
Butyl benzyl phthalate	81.1	11.7	45	115	35	130
Di-n-butyl phthalate	84.8	10.3	55	115	45	125
Di-n-octyl phthalate	87.4	16.6	35	135	20	155
Diethyl phthalate	79.2	12.9	40	120	30	130
Dimethyl phthalate	75.9	16.9	25	125	10	145
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	35	130	20	145
N-Nitrosodimethylamine	67.9	14.1	25	110	10	125
N-Nitrosodiphenylamine	79.6	10.6	50	110	35	120
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	76.2	10.2	45	105	35	115
Bis(2-chloroethyl) ether	73.3	12.3	35	110	25	120
Bis(2-chloroisopropyl) ether	78.2	17.5	25	130	10	150
Hexachlorobutadiene	65.2	12.6	25	105	15	115
Hexachloroethane	60.9	11.1	30	100	15	105

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846
METHOD 8270 WATER MATRIX¹⁴

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	35	105	25	120
1,2-Dichlorobenzene	67.3	11.4	35	100	20	115
1,3-Dichlorobenzene	64.8	10.9	30	100	20	110
1,4-Dichlorobenzene	64.8	10.9	30	100	20	110
2-Chloronaphthalene	76.5	9.3	50	105	40	115
4-Bromophenyl phenyl ether	82.9	10.2	50	115	40	125
4-Chlorophenyl phenyl ether	80.6	10.3	50	110	40	120
Hexachlorobenzene	82.3	10.0	50	110	40	120
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	50	120	40	130
2,6-Dinitrotoluene	82.7	11.3	50	115	35	130
2-Nitroaniline	81.8	11.2	50	115	35	125
3-Nitroaniline	72.6	17.7	20	125	10	145
4-Nitroaniline	77.2	13.7	35	120	20	130
Nitrobenzene	76.8	10.8	45	110	35	120
Neutral Aromatics						
Carbazole	82.5	11.4	50	115	35	130
Dibenzofuran	80.3	8.8	55	105	45	115
Others						
1,2-Diphenylhydrazine	84.8	9.4	55	115	45	120
Benzyl alcohol	71.0	13.8	30	110	15	125
Isophorone	81.0	10.5	50	110	40	125

TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS
SW-846 METHOD 8270 SOLID MATRIX¹⁵

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	45	105	35	115
Acenaphthene	77.3	10.3	45	110	35	120
Acenaphthylene	75.7	10.4	45	105	35	115
Anthracene	79.9	9.0	55	105	45	115

¹⁵ A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spike in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

**Table G-6. LCS Control Limits for Semivolatile Organic Compounds SW-846 Method 8270
Water Matrix⁵ (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	35	105	25	120
1,2-Dichlorobenzene	67.3	11.4	35	100	20	115
1,3-Dichlorobenzene	64.8	10.9	30	100	20	110
1,4-Dichlorobenzene	64.8	10.9	30	100	20	110
2-Chloronaphthalene	76.5	9.3	50	105	40	115
4-Bromophenyl phenyl ether	82.9	10.2	50	115	40	125
4-Chlorophenyl phenyl ether	80.6	10.3	50	110	40	120
Hexachlorobenzene	82.3	10.0	50	110	40	120
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	50	120	40	130
2,6-Dinitrotoluene	82.7	11.3	50	115	35	130
2-Nitroaniline	81.8	11.2	50	115	35	125
3-Nitroaniline	72.6	17.7	20	125	10	145
4-Nitroaniline	77.2	13.7	35	120	20	130
Nitrobenzene	76.8	10.8	45	110	35	120
Neutral Aromatics						
Carbazole	82.5	11.4	50	115	35	130
Dibenzofuran	80.3	8.8	55	105	45	115
Others						
1,2-Diphenylhydrazine	84.8	9.4	55	115	45	120
Benzyl alcohol	71.0	13.8	30	110	15	125
Isophorone	81.0	10.5	50	110	40	125

**Table G-7. LCS Control Limits for Semivolatile Organic Compounds
SW-846 Method 8270 Solid Matrix⁶**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	45	105	35	115
Acenaphthene	77.3	10.3	45	110	35	120
Acenaphthylene	75.7	10.4	45	105	35	115
Anthracene	79.9	9.0	55	105	45	115
Benz[a]anthracene	81.6	9.8	50	110	40	120
Benzo[a]pyrene	80.7	10.3	50	110	40	120
Benzo[b]fluoranthene	79.7	11.4	45	115	35	125
Benzo[k]fluoranthene	83.8	12.9	45	125	30	135
Benzo[g,h,i]perylene	81.8	14.7	40	125	25	140
Chrysene	82.6	9.9	55	110	45	120

⁶ A number of sporadic marginal exceedances (ME) of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section G.2 and Table G-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine. Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section G.5.

**TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS
SW-846 METHOD 8270 SOLID MATRIX¹⁵**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benz[a]anthracene	81.6	9.8	50	110	40	120
Benzo[a]pyrene	80.7	10.3	50	110	40	120
Benzo[b]fluoranthene	79.7	11.4	45	115	35	125
Benzo[k]fluoranthene	83.8	12.9	45	125	30	135
Benzo[g,h,i]perylene	81.8	14.7	40	125	25	140
Chrysene	82.6	9.9	55	110	45	120
Dibenz[a,h]anthracene	82.9	13.9	40	125	25	140
Fluoranthene	83.9	10.1	55	115	45	125
Fluorene	78.3	9.8	50	110	40	115
Indeno[1,2,3-cd]pyrene	79.7	13.8	40	120	25	135
Naphthalene	73.4	11.1	40	105	30	120
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	45	125	35	135
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	50	110	40	120
2,4,6-Trichlorophenol	76.3	11.0	45	110	30	120
2,4-Dichlorophenol	77.2	10.9	45	110	35	120
2,4-Dimethylphenol	67.3	11.9	30	105	20	115
2,4-Dinitrophenol	72.6	20.0	15	130	10	150
2-Chlorophenol	74.7	10.3	45	105	35	115
2-Methylphenol	71.7	10.6	40	105	30	115
2-Nitrophenol	76.2	11.5	40	110	30	120
3-Methylphenol/4-Methylphenol	73.9	10.9	40	105	30	120
4,6-Dinitro-2-methylphenol	83.1	18.0	30	135	10	155
4-Chloro-3-methylphenol	79.5	11.1	45	115	35	125
4-Nitrophenol	77.0	20.2	15	140	10	160
Pentachlorophenol	71.9	15.6	25	120	10	135
Phenol	69.7	10.2	40	100	30	110
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	87.4	13.3	45	125	35	140
Butyl benzyl phthalate	86.4	12.3	50	125	35	135
Di-n-butyl phthalate	83.2	9.1	55	110	45	120
Di-n-octyl phthalate	86.4	15.2	40	130	25	145
Diethyl phthalate	82.2	10.6	50	115	40	125
Dimethyl phthalate	79.6	10.2	50	110	40	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	115	30	125
N-Nitrosodimethylamine	66.1	15.9	20	115	10	130
N-Nitrosodiphenylamine	82.4	11.1	50	115	40	125
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	75.5	10.9	45	110	30	120
Bis(2-chloroethyl) ether	71.1	11.2	40	105	25	115
Bis(2-chloroisopropyl) ether	68.4	15.7	20	115	10	130

**Table G-7. LCS Control Limits for Semivolatile Organic Compounds
SW-846 Method 8270 Solid Matrix⁶ (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Dibenz[a,h]anthracene	82.9	13.9	40	125	25	140
Fluoranthene	83.9	10.1	55	115	45	125
Fluorene	78.3	9.8	50	110	40	115
Indeno[1,2,3-cd]pyrene	79.7	13.8	40	120	25	135
Naphthalene	73.4	11.1	40	105	30	120
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	45	125	35	135
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	50	110	40	120
2,4,6-Trichlorophenol	76.3	11.0	45	110	30	120
2,4-Dichlorophenol	77.2	10.9	45	110	35	120
2,4-Dimethylphenol	67.3	11.9	30	105	20	115
2,4-Dinitrophenol	72.6	20.0	15	130	10	150
2-Chlorophenol	74.7	10.3	45	105	35	115
2-Methylphenol	71.7	10.6	40	105	30	115
2-Nitrophenol	76.2	11.5	40	110	30	120
3-Methylphenol/4-Methylphenol	73.9	10.9	40	105	30	120
4,6-Dinitro-2-methylphenol	83.1	18.0	30	135	10	155
4-Chloro-3-methylphenol	79.5	11.1	45	115	35	125
4-Nitrophenol	77.0	20.2	15	140	10	160
Pentachlorophenol	71.9	15.6	25	120	10	135
Phenol	69.7	10.2	40	100	30	110
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	87.4	13.3	45	125	35	140
Butyl benzyl phthalate	86.4	12.3	50	125	35	135
Di-n-butyl phthalate	83.2	9.1	55	110	45	120
Di-n-octyl phthalate	86.4	15.2	40	130	25	145
Diethyl phthalate	82.2	10.6	50	115	40	125
Dimethyl phthalate	79.6	10.2	50	110	40	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	115	30	125
N-Nitrosodimethylamine	66.1	15.9	20	115	10	130
N-Nitrosodiphenylamine	82.4	11.1	50	115	40	125
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	75.5	10.9	45	110	30	120
Bis(2-chloroethyl) ether	71.1	11.2	40	105	25	115
Bis(2-chloroisopropyl) ether	68.4	15.7	20	115	10	130
Hexachlorobutadiene	78.2	12.9	40	115	25	130
Hexachloroethane	71.9	12.6	35	110	20	120
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	45	110	30	120
1,2-Dichlorobenzene	70.9	8.7	45	100	35	105
1,3-Dichlorobenzene	69.7	10.3	40	100	30	110
1,4-Dichlorobenzene	69.0	11.4	35	105	25	115

**TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS
SW-846 METHOD 8270 SOLID MATRIX¹⁶**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Hexachlorobutadiene	78.2	12.9	40	115	25	130
Hexachloroethane	71.9	12.6	35	110	20	120
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	45	110	30	120
1,2-Dichlorobenzene	70.9	8.7	45	95	35	105
1,3-Dichlorobenzene	69.7	10.3	40	100	30	110
1,4-Dichlorobenzene	69.0	11.4	35	105	25	115
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	45	115	35	130
4-Chlorophenyl phenyl ether	79.6	10.7	45	110	35	120
Hexachlorobenzene	82.5	11.7	45	120	35	130
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	50	115	35	130
2,6-Dinitrotoluene	80.2	10.7	50	110	35	125
2-Nitroaniline	81.0	12.2	45	120	30	130
3-Nitroaniline	68.8	13.8	25	110	15	125
4-Nitroaniline	73.6	13.1	35	115	20	125
Nitrobenzene	77.2	11.9	40	115	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	45	115	30	130
Dibenzofuran	77.1	8.8	50	105	40	110
Others						
Benzyl alcohol	70.9	17.4	20	125	10	140
Isophorone	77.0	11.4	45	110	30	125

**TABLE D-8. LCS CONTROL LIMITS FOR CHLORINATED HERBICIDES SW-846
METHOD 8151 WATER MATRIX¹⁶**

Analyte	Median	Lower Control Limit	Upper Control Limit
2,4-D	88	35	115
2,4-DB	99	45	130
2,4,5-T	83	35	110
2,4,5-TP (Silvex)	87	50	115
Dalapon	62	40	110
Dicamba	86	60	110
Dichloroprop	91	70	120
Dinoseb	65	20	95
MCPA	93	60	145

¹⁶ LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for MCPA, although the compound does appear on the target analyte list for method 8151 (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

**Table G-7. LCS Control Limits for Semivolatile Organic Compounds
SW-846 Method 8270 Solid Matrix (continued)**

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	45	115	35	130
4-Chlorophenyl phenyl ether	79.6	10.7	45	110	35	120
Hexachlorobenzene	82.5	11.7	45	120	35	130
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	50	115	35	130
2,6-Dinitrotoluene	80.2	10.7	50	110	35	125
2-Nitroaniline	81.0	12.2	45	120	30	130
3-Nitroaniline	68.8	13.8	25	110	15	125
4-Nitroaniline	73.6	13.1	35	115	20	125
Nitrobenzene	77.2	11.9	40	115	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	45	115	30	130
Dibenzofuran	77.1	8.8	50	105	40	110
Others						
Benzyl alcohol	70.9	17.4	20	125	10	140
Isophorone	77.0	11.4	45	110	30	125

Table G-8. LCS Control Limits for Chlorinated Herbicides SW-846 Method 8151 Water Matrix⁷

Analyte	Median	Lower Control Limit	Upper Control Limit
2,4-D	88	35	115
2,4-DB	99	45	130
2,4,5-T	83	35	110
2,4,5-TP (Silvex)	87	50	115
Dalapon	62	40	110
Dicamba	86	60	110
Dichloroprop	91	70	120
Dinoseb	65	20	100
MCPA	93	60	145

⁷ LCS control limits were generated using non-parametric statistics (see section G.1 for further explanation). LCS control limits are not available for MCPA. Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

Attachment 3

Attachment 1 Corrective Action and Documentation Examples

<u>OCCURRENCE</u>	<u>ACTION</u>	<u>DOCUMENTATION</u>
1. Initial calibration does not meet QC criteria.	1. Investigate source of problem, determine if source is an instrument problem or a standard solution problem. If problem is with a single point of the ICAL, reanalyze the bad standard and reevaluate. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	1. Notation in instrument run log book, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification check does not meet QC criteria.	2. Investigate source of problem, determine if source is with ICAL or ICV, is it an instrument problem or a standard solution problem, reanalyze ICV or perform new ICAL.	2. Notation in instrument run log book, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria.	3. Investigate source of problem. If source is instrument, perform instrument maintenance and reanalyze CCV. If CCV still will not pass, repeat the above, or perform new initial calibration. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	3. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
4. GC/MS tune does not meet method criteria.	4. Investigate source of problem, evaluate instrument response to cal gas (PFTBA), when instrument response to PFTBA is improved, re-inject DFTPP tune.	4. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
5. Method blank contains target compound above reporting limit.	5. Investigate source of problem. Reanalyze all effected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released with commentary.	5. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.

<p>5. Surrogate standard outside of acceptable range.</p>	<p>6. Investigate source of problem. If it is determined to be an instrument problem, reanalyze sample. If it is determined to be a preparation problem, reextract/reanalyze the sample. If it can be determined to be an obvious matrix problem (masking of surrogate by target or non-target compound at significantly greater concentration, excessive hydrocarbons in sample, other knowledge of sample matrix, etc.) the sample may be reanalyzed at dilution to reduce interference or reported with notation in narrative, depending on project objectives.</p>	<p>6. If only reanalysis is reported, notation in instrument run log. If both sets of data are to be reported, notation in instrument run log, reextraction request form, preparation logbooks, commentary on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. If source of problem found to be systematic (bad spike solution, etc). a formal corrective action form must be initiated.</p>
<p>7. Compound out of acceptance range in laboratory control sample.</p>	<p>7. Investigate source of problem. If LCS is acceptable per method/SOP specifications, associated sample data can be reported. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP requirements, re-extract all associated samples. If insufficient sample volume, notify project manager to discuss with client, report initial data if no other sample can be provided.</p>	<p>7. If LCS is acceptable per method/SOP, flag all compounds out of range on Form 3 of data report, if samples are reanalyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook and commentary in data review checklist to be included in project narrative. If reanalysis cannot be performed due to insufficient sample, commentary in data review checklist to be included in project narrative. If source of problem found to be systematic (bad spike solution, etc). a formal corrective action form must be initiated.</p>
<p>3. Compound in sample exceeds upper calibration standard concentration.</p>	<p>8. Reanalyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running instrument using autosampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is</p>	<p>8. Notation in instrument run log book. If initial analysis is reported, flag compound exceeding calibration limit with "E" on data report and commentary on data review checklist to be included in project narrative. If only diluted analysis is to be reported, commentary in data review checklist to be included in project narrative. If both initial and dilution are to be reported, all of the above.</p>

<p>Instrument blank (GC) contains contamination above QC criteria.</p> <p>Matrix spike recovery out of QC range.</p> <p>Duplicate (or MSD) relative percent difference exceeds QC limit.</p> <p>Internal Standard areas exceed QC criteria. (-50% to +100%)</p> <p>A. CCV. B. QC (blank, LCS) C. Samples and MS/MSD.</p>	<p>required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be reanalyzed if sufficient volume exists.</p> <p>9. Investigate source of problem, decontaminate purge and trap instrument, reanalyze all effected samples.</p> <p>10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in unspiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, reanalyze spike sample.</p> <p>11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, reanalyze duplicate sample.</p> <p>12. A. Evaluate problem. Re-analyze CCV. If the CCV does not meet criteria, re-analyze the initial calibration and proceed with CCV/QC/samples. B. Evaluate CCV and QC. As blank and LCS are "interference-free matrix" the IS areas should be within the same limits as the CCV. Evaluate for potential problems. If time allows (data not required on a rush basis), reanalyze QC prior to sample analysis. If insufficient time due to client deadline, data may be reported (as they meet method requirements) but the issue should be noted for the data</p>	<p>Notation in instrument run logbook, and if instrument maintenance performed, in instrument maintenance logbook.</p> <p>Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p> <p>Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.</p> <p>A. Document in the analytical run log. B. If the criteria are met after re-analysis, document in run log. If the criteria has not been met and the results of the sample batch is reported, document in the run log and the Corrective Action Logbook. Have the supervisor review situation, initial/date, and include a comment on the data review checklist for the data reviewer and for inclusion in the narrative information to the client. C. If the CCV and QC meet criteria, document in the run</p>
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reviewer and for the client.

C. Evaluate CCV and QC. The IS areas may indicate a potential problem, or matrix interference. If the CCV and batch QC meet criteria, document and report results as matrix interference. In particular, if the recovery of the surrogate standard associated with the IS compound is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery criteria, the IS issue is impacting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias) This requires notation and communication of the effect to the data reviewer and the client. Based on the severity of the problem, discuss with supervisor, technical director and /or reanalyze effected samples. If results are reported as is, document per 12C.

log and on the package checklist for inclusion in the narrative submitted to the client. Note that certain compounds may be potentially high bias or potentially low bias due to IS recoveries outside of range. If QC and samples do not meet criteria and the results are reported, document in the run log, document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

Attachment 4



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with DFTPP	Inter-laboratory consistency and comparability	(1) Criteria for DFTPP listed in Table 3 of SW-846 8270C (the same criteria must be used for all analyses) (2) Every 12 hours (3) DDT breakdown should be evaluated and should be <20%. (4) Pentachlorophenol and benzidine peak tailing should be evaluated. Peak tailing factor must be <3 for benzidine and <5 for pentachlorophenol. NOTE: Tune must be performed in full scan mode for SIM analyses.	No	Perform instrument/injection port maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified. Report DDT breakdown and peak tailing exceedances in the case narrative.
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be \leq reporting limit (3) Full scan: %RSD should be ≤ 15 or "r" should be ≥ 0.99 for all compounds except CCCs which must be ≤ 30 % RSD or "r" ≥ 0.99 SIM: %RSD should be ≤ 20 or "r" should be ≥ 0.99 for all compounds (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin. (6) SIM: Laboratory must monitor a minimum of two ions per analyte (the primary ion or quantitation ion and a minimum of one confirmation ion); this is required for all target analytes, surrogates and internal standards	No	Recalibrate as required by method (1) if any of CCC %RSDs >30 or any of CCC "r" <0.99 or (2) if >20% of remaining analytes have %RSDs >30 or "r" <0.99.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) Full scan: Percent difference or percent drift must be ≤ 20 for CCCs and should be ≤ 30 for other compounds SIM: Percent difference or percent drift should be ≤ 30 for all compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20 or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in case narrative.



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	<ol style="list-style-type: none"> (1) Extracted with every batch or every 20 samples, whichever is more frequent (2) Matrix-specific (e.g., water, soil) (3) Target analytes must be < RL except for common laboratory contaminants (such as phthalates) which must be <5x RL 	Yes	Locate source of contamination; correct problem; re-extract associated samples if uncommon contaminants are present in the method blank.	<ol style="list-style-type: none"> (1) Report non-conformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the case narrative. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	<ol style="list-style-type: none"> (1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level should be between low and mid-level standard (4) Must contain all target analytes (5) Matrix-specific (e.g., soil, water) (6) Percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above. 	Yes	Recalculate the percent recoveries; Re-extract associated samples if >20% of all analytes fall outside the acceptance criteria or if >15% of analytes from a particular class (base-neutral or acid) fall outside the acceptance criteria.	<ol style="list-style-type: none"> (1) Report non-conformances in case narrative. (2) Individual laboratories should identify and document "difficult" (***) analytes for which laboratory-determined recovery ranges routinely exceed the 100 ± 30% criterion. Exceedances for these "difficult" analytes should be qualified in case narrative. Analytical data to support the "difficult" analyte classification are to be available for review during an audit. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Method Precision	<ol style="list-style-type: none"> (1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory-determined percent recoveries must be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds (6) Matrix-specific (e.g., soil, water, etc.) (7) Laboratory-determined Relative Percent Difference (RPD) must be ≤20 for waters and ≤30 for solids except for "difficult" (***) analytes which must be ≤ 50. 	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	<ol style="list-style-type: none"> (1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (***) analytes for which laboratory-determined RPDs routinely exceed the ≤ 25 criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level should be between low and mid-level standard (5) Must contain all target analytes. (6) Percent recoveries should be between 40 – 140 for the base-neutral compounds and between 30 -130 for the acid compounds, or develop laboratory in-house limits. (7) RPDs should be ≤20 for waters and ≤30 for solids	Yes Only when requested by the data-user,	Check LCS; if recoveries acceptable in LCS, evaluate alternate cleanup techniques for samples associated with MS/MSD and/or narrate non-conformance.	Note exceedances in case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Minimum of 3 base-neutral and 3 acid, at retention times across GC run Recommended base-neutral surrogates: nitrobenzene-d5, 2-fluorobiphenyl, terphenyl-d14 Recommended acid surrogates: phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol <i>SIM Note:</i> Surrogates used must be representative of compound class of target analytes (e.g., use base-neutral surrogates if analyzing for PAHs, use acid surrogates if analyzing for pentachlorophenol). (2) Percent recoveries in soil must be between 30-130 for all surrogates. Percent recoveries in water must be between 30-130 for base-neutral surrogates and between 15-110 for acid surrogates. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	If two or more surrogates for any one fraction (base-neutral or acid) are outside limits or if any one surrogate recovers at <10%, reextract the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.	(1) Note exceedances in case narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory should report results of both extractions. (3) If re-extraction is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction. (5) If sample is not re-extracted due to obvious interference, the laboratory must provide the chromatogram in the data report.



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) <i>Full scan</i> : Minimum of 6 at retention times across GC run. <i>SIM</i> : Number of internal standards used will be dependent on the analytes of interest. Internal standards must elute in close proximity to the analytes of interest. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard (Section 5.4.2 of 8270C) (3) Retention times of internal standards must be within ± 30 seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, re-analyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in case narrative. (2) If re-analysis yields similar internal standard non-conformances, the laboratory should report both results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on internal standard calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte (3) The internal standard used for quantitation shall be the one nearest the retention time of the subject analyte.	NA	NA	If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the case narrative with a list of the affected analytes.



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting Issues	NA	(1) The laboratory must only report values \geq the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported (3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance			(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit. (3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II B-3. (4) The performance of dilutions must be documented in the case narrative.

GC/MS = Gas Chromatography/Mass Spectrometry
 DF TPP = Decafluorotriphenylphosphine
 MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
 %RSD = Percent Relative Standard Deviation
 UCM = Unresolved Complex Mixture

"r" = Correlation Coefficient
 CCCs = Calibration Check Compounds
 RPDs = Relative Percent Differences
 TIC = Tentatively Identified Compound
 NA = Not Applicable

Potentially "difficult" analytes include: dimethyl phthalate, 4-nitrophenol, phenol, 4-methylphenol, 2-methylphenol, 2,4-dinitrophenol, pentachlorophenol, and 4-chloroaniline

APPENDIX H
DETERMINATION OF PROJECT ACTION LIMITS

**TABLE H-1
DETERMINATION OF PROJECT SCREENING LEVELS
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND**

Analyte	CAS Number	A		B		C		D		Lower of EPA RSL Residential Soil or Sediment PSL (Column C or D) = Residual Material PSL ⁽⁴⁾ (mg/kg)	Lower of Confirmatory Soil PSL or Sediment PSL Reference ⁽⁴⁾	RIDEM DEC - Residential Soil (mg/kg) ⁽⁵⁾
		EPA RSL Residential Soil ⁽¹⁾ = Test Pit Soil PSL (mg/kg)		Selected Ecological Soil Screening Level (SSL) ⁽²⁾ (mg/kg)		Selected Ecological SSL Reference ⁽²⁾		Lower of EPA RSL Residential Soil or Ecological SSL (Column A or B) = Confirmatory Soil PSL (mg/kg)	Confirmatory Soil PSL Reference			
Volatile Organic Compounds												
1,1,1-Trichloroethane	71-55-6	870	N	29.8	Region 5 ESL	29.8	Region 5 ESL	0.0302	Region 3 BTAG	0.0302	Sed PSL	540
1,1,2,2-Tetrachloroethane	79-34-5	0.56	C	0.127	Region 5 ESL	0.127	Region 5 ESL	1.36	Region 3 BTAG	0.127	CS PSL	1.3
1,1,2-Trichloroethane	79-00-5	1.1	C	28.6	Region 5 ESL	1.1	EPA RSL Res	1.24	Region 3 BTAG	1.1	CS PSL	3.6
1,1-Dichloroethane	75-34-3	3.3	C	20.1	Region 5 ESL	3.3	EPA RSL Res	0.027	SCV	0.027	Sed PSL	920
1,1-Dichloroethene	75-35-4	24	N	8.28	Region 5 ESL	8.28	Region 5 ESL	0.031	Region 3 BTAG	0.031	Sed PSL	0.2
1,2-Dichlorobenzene	95-50-1	190	N	2.96	Region 5 ESL	2.96	Region 5 ESL	0.0165	Region 3 BTAG	0.0165	Sed PSL	510
1,2-Dichloroethane	107-06-2	0.43	C	21.2	Region 5 ESL	0.43	EPA RSL Res	0.25	SCV	0.25	Sed PSL	0.9
1,2-Dichloropropane	78-87-5	0.89	C	32.7	Region 5 ESL	0.89	EPA RSL Res	--	--	0.89	CS PSL	1.9
1,2,4-Trichlorobenzene	120-82-1	6.2	N	11.1	Region 5 ESL	6.2	EPA RSL Res	2.1	Region 3 BTAG	2.1	Sed PSL	96
1,2,4-Trimethylbenzene	95-63-6	6.2	N	--	--	6.2	EPA RSL Res	--	--	6.2	CS PSL	--
1,3,5-Trimethylbenzene	108-67-8	78	N	--	--	78	EPA RSL Res	--	--	78	CS PSL	--
1,3-Dichlorobenzene	541-73-1	--	--	37.7	Region 5 ESL	37.7	Region 5 ESL	4.43	Region 3 BTAG	4.43	Sed PSL	430
1,4-Dichlorobenzene	106-46-7	2.4	C	0.546	Region 5 ESL	0.546	Region 5 ESL	0.599	Region 3 BTAG	0.546	CS PSL	27
2-Butanone (MEK)	78-93-3	2800	N	89.6	Region 5 ESL	89.6	Region 5 ESL	0.27	SCV	0.27	Sed PSL	10000
2-Hexanone	591-78-6	21	N	12.6	Region 5 ESL	12.6	Region 5 ESL	0.022	SCV	0.022	Sed PSL	-
4-Methyl-2-pentanone (MIBK)	108-10-1	530	N	443	Region 5 ESL	443	Region 5 ESL	0.033	SCV	0.033	Sed PSL	1200
Acetone	67-64-1	6100	N	2.5	Region 5 ESL	2.5	Region 5 ESL	0.0087	SCV	0.0087	Sed PSL	7800
Benzene	71-43-2	1.1	C	0.255	Region 5 ESL	0.255	Region 5 ESL	0.16	SCV	0.16	Sed PSL	2.5
Bromodichloromethane	75-27-4	0.27	C	0.54	Region 5 ESL	0.27	EPA RSL Res	--	--	0.27	CS PSL	10
Bromoform	75-25-2	61	C	15.9	Region 5 ESL	15.9	Region 5 ESL	0.654	Region 3 BTAG	0.654	Sed PSL	81
Bromomethane	74-83-9	0.73	N	0.235	Region 5 ESL	0.235	Region 5 ESL	--	--	0.235	CS PSL	0.8
Carbon Disulfide	75-15-0	82	N	0.0941	Region 5 ESL	0.0941	Region 5 ESL	0.000851	Region 3 BTAG	0.000851	Sed PSL	-
Carbon Tetrachloride	56-23-5	0.61	C	2.98	Region 5 ESL	0.61	EPA RSL Res	0.0642	Region 3 BTAG	0.0642	Sed PSL	1.5
Chlorobenzene	108-90-7	29	N	13.1	Region 5 ESL	13.1	Region 5 ESL	0.00842	Region 3 BTAG	0.00842	Sed PSL	210
Chloroethane	75-00-3	1500	N	--	--	1500	EPA RSL Res	--	--	1500	CS PSL	-
Chloroform	67-66-3	0.29	C	1.19	Region 5 ESL	0.29	EPA RSL Res	0.022	SCV	0.022	Sed PSL	1.2
Chloromethane	74-87-3	12	N	10.4	Region 5 ESL	10.4	Region 5 ESL	--	--	10.4	CS PSL	-
cis-1,2-Dichloroethene	156-59-2	78	N	0.78373	Region 5 ESL	0.78373	Region 5 ESL	0.4	SCV	0.4	Sed PSL	630
cis-1,3-Dichloropropene	10061-01-5	1.7	C	0.398	Region 5 ESL	0.398	Region 5 ESL	0.000051	SCV	0.000051	Sed PSL	-
Dibromochloromethane	124-48-1	0.68	C	2.05	Region 5 ESL	0.68	EPA RSL Res	--	--	0.68	CS PSL	7.6
Ethylbenzene	100-41-4	5.4	C	5.16	Region 5 ESL	5.16	Region 5 ESL	1.1	Region 3 BTAG	1.1	Sed PSL	71
Methylene Chloride	75-09-2	11	C	4.05	Region 5 ESL	4.05	Region 5 ESL	0.37	SCV	0.37	Sed PSL	45
Styrene	100-42-5	630	N	4.69	Region 5 ESL	4.69	Region 5 ESL	0.559	Region 3 BTAG	0.559	Sed PSL	13
Tetrachloroethene	127-18-4	0.55	C	9.92	Region 5 ESL	0.55	EPA RSL Res	0.468	Region 3 BTAG	0.468	Sed PSL	12
Toluene	108-88-3	500	N	5.45	Region 5 ESL	5.45	Region 5 ESL	0.05	SCV	0.05	Sed PSL	190
trans-1,2-Dichloroethene	156-60-5	15	N	0.784	Region 5 ESL	0.784	Region 5 ESL	1.05	Region 3 BTAG	0.784	CS PSL	1100
trans-1,3-Dichloropropene	10061-02-6	1.7	C	0.398	Region 5 ESL	0.398	Region 5 ESL	0.000051	SCV	0.000051	Sed PSL	-
Trichloroethene	79-01-6	2.8	C	12.4	Region 5 ESL	2.8	EPA RSL Res	0.0969	Region 3 BTAG	0.0969	Sed PSL	13
Trichlorofluoromethane (CFC-11)	75-69-4	79	N	16.4	Region 5 ESL	16.4	Region 5 ESL	--	--	16.4	CS PSL	-
Vinyl Chloride	75-01-4	0.06	C	0.646	Region 5 ESL	0.06	EPA RSL Res	--	--	0.06	CS PSL	0.02
Xylenes (total)	1330-20-7	63	N	10	Region 5 ESL	10	Region 5 ESL	0.16	SCV	0.16	Sed PSL	110
Semivolatile Organic Compounds												
2,4,5-Trichlorophenol	95-95-4	610	N	4	Plant ORNL	4	ORNL Plant	0.003	NOAA SQuiRT	0.003	Sed PSL	330
2,4,6-Trichlorophenol	88-06-2	6.1	N	9.94	Region 5 ESL	6.1	EPA RSL Res	0.213	Region 3 BTAG	0.213	Sed PSL	58
2,4-Dichlorophenol	120-83-2	18	N	87.5	Region 5 ESL	18	EPA RSL Res	0.117	Region 3 BTAG	0.117	Sed PSL	30
2,4-Dimethylphenol	105-67-9	120	N	0.01	Region 5 ESL	0.01	Region 5 ESL	0.029	Region 3 BTAG	0.01	CS PSL	1400
2,4-Dinitrophenol	51-28-5	12	N	0.0609	Region 5 ESL	0.0609	Region 5 ESL	--	--	0.0609	CS PSL	160
2,4-Dinitrotoluene	121-14-2	1.6	C	1.28	Region 5 ESL	1.28	Region 5 ESL	0.0416	Region 3 BTAG	0.0416	Sed PSL	0.9
2,6-Dinitrotoluene	606-20-2	6.1	N	0.0328	Region 5 ESL	0.0328	Region 5 ESL	--	--	0.0328	CS PSL	-
2-Chloronaphthalene	91-58-7	630	N	0.0122	Region 5 ESL	0.0122	Region 5 ESL	--	--	0.0122	CS PSL	-
2-Chlorophenol	95-57-8	39	N	0.243	Region 5 ESL	0.243	Region 5 ESL	0.0312	Region 3 BTAG	0.0312	Sed PSL	50
2-Methylphenol	95-48-7	310	N	40.4	Region 5 ESL	40.4	Region 5 ESL	0.012	SCV	0.012	Sed PSL	-
2-Nitroaniline	88-74-4	61	N	74.1	Region 5 ESL	61	EPA RSL Res	--	--	61	CS PSL	-
2-Nitrophenol	88-75-5	12	N	1.6	Region 5 ESL	1.6	Region 5 ESL	--	--	1.6	CS PSL	-
3,3'-Dichlorobenzidine	91-94-1	1.1	C	0.646	Region 5 ESL	0.646	Region 5 ESL	0.127	Region 3 BTAG	0.127	Sed PSL	1.4
3-Nitroaniline	99-09-2	--	--	3.16	Region 5 ESL	3.16	Region 5 ESL	--	--	3.16	CS PSL	-
4,6-Dinitro-2-methylphenol	534-52-1	0.49	N	0.144	Region 5 ESL	0.144	Region 5 ESL	--	--	0.144	CS PSL	-

TABLE H-1
DETERMINATION OF PROJECT SCREENING LEVELS
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS Number	A		B		C		D		RIDEDEC Residential Soil (mg/kg) ⁽⁵⁾		
		EPA RSL Residential Soil ⁽¹⁾ = Test Pit Soil PSL (mg/kg)		Selected Ecological Soil Screening Level (SSL) ⁽²⁾ (mg/kg)	Selected Ecological SSL Reference ⁽²⁾	Lower of EPA RSL Residential Soil or Ecological SSL (Column A or B) = Confirmatory Soil PSL (mg/kg)	Confirmatory Soil PSL Reference	Ecological Sediment Screening Level ⁽³⁾ = Sediment PSL (mg/kg)	Sediment PSL Reference ⁽³⁾		Lower of Confirmatory Soil PSL or Sediment PSL (Column C or D) = Residual Material PSL ⁽⁴⁾ (mg/kg)	Lower of Confirmatory Soil PSL or Sediment PSL Reference ⁽⁴⁾
4-Bromophenyl phenyl ether	101-55-3	--	--	--	--	--	--	1.23	Region 3 BTAG	1.23	Sed PSL	-
4-Chloro-3-methylphenol	59-50-7	610	N	7.95	Region 5 ESL	7.95	Region 5 ESL	--	--	7.95	CS PSL	-
4-Chloroaniline	106-47-8	2.4	C	1.1	Region 5 ESL	1.1	Region 5 ESL	--	--	1.1	CS PSL	310
4-Chlorophenyl phenyl ether	7005-72-3	--	--	--	--	--	--	--	--	--	--	-
4-Methylphenol	106-44-5	31	N	163	Region 5 ESL	31	EPA RSL Res	0.67	Region 3 BTAG	0.67	Sed PSL	-
4-Nitroaniline	100-01-6	24	C	21.9	Region 5 ESL	21.9	Region 5 ESL	--	--	21.9	CS PSL	-
4-Nitrophenol	100-02-7	--	--	5.12	Region 5 ESL	5.12	Region 5 ESL	--	--	5.12	CS PSL	-
Bis(2-Chloroethoxy)methane	111-91-1	18	N	0.302	Region 5 ESL	0.302	Region 5 ESL	--	--	0.302	CS PSL	-
bis(2-Chloroethyl)ether	111-44-4	0.21	C	23.7	Region 5 ESL	0.21	EPA RSL Res	--	--	0.21	CS PSL	0.6
Bis(2-Ethylhexyl)phthalate	117-81-7	35	C	0.925	Region 5 ESL	0.925	Region 5 ESL	0.18	Region 3 BTAG	0.18	Sed PSL	46
Butyl benzyl phthalate	85-68-7	260	C	0.239	Region 5 ESL	0.239	Region 5 ESL	10.9	Region 3 BTAG	0.239	CS PSL	-
Carbazole	86-74-8	--	--	--	--	--	--	--	--	--	--	-
Dibenzofuran	132-64-9	7.8	N	--	--	7.8	EPA RSL Res	0.415	Region 3 BTAG	0.415	Sed PSL	-
Diethyl phthalate	84-66-2	4900	N	24.8	Region 5 ESL	24.8	Region 5 ESL	0.603	Region 3 BTAG	0.603	Sed PSL	340
Dimethyl phthalate	131-11-3	4900	N	734	Region 5 ESL	734	Region 5 ESL	0.006	NOAA SQuiRT	0.006	Sed PSL	1900
Di-n-butyl phthalate	84-74-2	610	N	0.15	Region 5 ESL	0.15	Region 5 ESL	6.47	Region 3 BTAG	0.15	CS PSL	-
Di-n-octyl phthalate	117-84-0	--	--	709	Region 5 ESL	709	Region 5 ESL	0.061	NOAA SQuiRT	0.061	Sed PSL	-
Hexachlorobutadiene	87-68-3	6.1	N	0.0398	Region 5 ESL	0.0398	Region 5 ESL	0.0013	NOAA SQuiRT	0.0013	Sed PSL	8.2
Hexachlorocyclopentadiene	77-47-4	37	N	0.755	Region 5 ESL	0.755	Region 5 ESL	--	--	0.755	CS PSL	-
Hexachloroethane	67-72-1	6.1	N	0.596	Region 5 ESL	0.596	Region 5 ESL	1.027	Region 3 BTAG	0.596	CS PSL	46
Isophorone	78-59-1	510	C	139	Region 5 ESL	139	Region 5 ESL	--	--	139	CS PSL	-
Nitrobenzene	98-95-3	4.8	C	1.31	Region 5 ESL	1.31	Region 5 ESL	0.021	NOAA SQuiRT	0.021	Sed PSL	-
N-Nitrosodi-n-propylamine	621-64-7	0.069	C	0.544	Region 5 ESL	0.069	EPA RSL Res	--	--	0.069	CS PSL	-
N-Nitrosodiphenylamine	86-30-6	99	C	0.545	Region 5 ESL	0.545	Region 5 ESL	2.68	Region 3 BTAG	0.545	CS PSL	-
Pentachlorophenol	87-86-5	3	C	2.1	Wildlife EPA SSL	2.1	EPA SSL Wildlife	0.504	Region 3 BTAG	0.504	Sed PSL	5.3
Phenol	108-95-2	1800	N	30	Invert ORNL	30	ORNL Invert	0.42	Region 3 BTAG	0.42	Sed PSL	6000
Polycyclic Aromatic Hydrocarbons												
2-Methylnaphthalene	91-57-6	31	N	29	Invert EPA SSL	29	EPA SSL Invert	0.0202	Region 3 BTAG	0.0202	Sed PSL	123
Acenaphthene	83-32-9	340	N	20	Plant ORNL	20	ORNL Plant	0.0067	Region 3 BTAG	0.0067	Sed PSL	43
Acenaphthylene	208-96-8	340	N	29	Invert EPA SSL	29	EPA SSL Invert	0.0059	Region 3 BTAG	0.0059	Sed PSL	23
Anthracene	120-12-7	1700	N	29	Invert EPA SSL	29	EPA SSL Invert	0.0572	Region 3 BTAG	0.0572	Sed PSL	35
Benzo(a)anthracene	56-55-3	0.15	C	1.1	Wildlife EPA SSL	0.15	EPA RSL Res	0.108	Region 3 BTAG	0.108	Sed PSL	0.9
Benzo(a)pyrene	50-32-8	0.015	C	1.1	Wildlife EPA SSL	0.015	EPA RSL Res	0.15	Region 3 BTAG	0.015	CS PSL	0.4
Benzo(b)fluoranthene	205-99-2	0.15	C	1.1	Wildlife EPA SSL	0.15	EPA RSL Res	0.13	NOAA SQuiRT	0.13	Sed PSL	0.9
Benzo(g,h,i)perylene	191-24-2	170	N	1.1	Wildlife EPA SSL	1.1	EPA SSL Wildlife	0.17	Region 3 BTAG	0.17	Sed PSL	0.8
Benzo(k)fluoranthene	207-08-9	1.5	C	1.1	Wildlife EPA SSL	1.1	EPA SSL Wildlife	0.24	Region 3 BTAG	0.24	Sed PSL	0.9
Chrysene	218-01-9	15	C	1.1	Wildlife EPA SSL	1.1	EPA SSL Wildlife	0.166	Region 3 BTAG	0.166	Sed PSL	0.4
Dibenzo(a,h)anthracene	53-70-3	0.015	C	1.1	Wildlife EPA SSL	0.015	EPA RSL Res	0.033	Region 3 BTAG	0.015	CS PSL	0.4
Fluoranthene	206-44-0	230	N	29	Invert EPA SSL	29	EPA SSL Invert	0.423	Region 3 BTAG	0.423	Sed PSL	20
Fluorene	86-73-7	230	N	29	Invert EPA SSL	29	EPA SSL Invert	0.0774	Region 3 BTAG	0.0774	Sed PSL	28
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	C	1.1	Wildlife EPA SSL	0.15	EPA RSL Res	0.017	Region 3 BTAG	0.017	Sed PSL	0.9
Naphthalene	91-20-3	3.6	C	29	Invert EPA SSL	3.6	EPA RSL Res	0.176	Region 3 BTAG	0.176	Sed PSL	54
Phenanthrene	85-01-8	170	N	29	Invert EPA SSL	29	EPA SSL Invert	0.204	Region 3 BTAG	0.204	Sed PSL	40
Pyrene	129-00-0	170	N	1.1	Wildlife EPA SSL	1.1	EPA SSL Wildlife	0.195	Region 3 BTAG	0.195	Sed PSL	13
Polychlorinated Biphenyls (PCBs)												
Aroclor-1016	12674-11-2	0.39	N	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1221	11104-28-2	0.14	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1232	11141-16-5	0.14	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1242	53469-21-9	0.22	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1248	12672-29-6	0.22	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1254	11097-69-1	0.11	N	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1260	11096-82-5	0.22	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1262	37324-23-5	0.22	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
Aroclor-1268	11100-14-4	0.22	C	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	-
PCBs (Total)	--	--	--	0.000332	Region 5 ESL	0.000332	Region 5 ESL	0.0598	Region 3 BTAG	0.000332	CS PSL	10
Metals												
Aluminum	7429-90-5	7700	N	50	Plant ORNL	50	ORNL Plant	25500	NOAA SQuiRT	50	CS PSL	-
Antimony	7440-36-0	3.1	N	0.27	Wildlife EPA SSL	0.27	EPA SSL Wildlife	2	Region 3 BTAG	0.27	CS PSL	10

**TABLE H-1
DETERMINATION OF PROJECT SCREENING LEVELS
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND**

Analyte	CAS Number	A		B		C		D		Lower of Confirmatory Soil PSL or Sediment PSL (Column C or D) = Residual Material PSL ⁽⁴⁾ (mg/kg)	Lower of Confirmatory Soil PSL or Sediment PSL Reference ⁽⁴⁾	RIDEM DEC - Residential Soil (mg/kg) ⁽⁵⁾
		EPA RSL Residential Soil ⁽¹⁾ = Test Pit Soil PSL (mg/kg)		Selected Ecological Soil Screening Level (SSL) ⁽²⁾ (mg/kg)	Selected Ecological SSL Reference ⁽²⁾	Lower of EPA RSL Residential Soil or Ecological SSL (Column A or B) = Confirmatory Soil PSL (mg/kg)	Confirmatory Soil PSL Reference	Ecological Sediment Screening Level ⁽³⁾ = Sediment PSL (mg/kg)	Sediment PSL Reference ⁽³⁾			
Arsenic	7440-38-2	0.39	C	18	Plant EPA SSL	0.39	EPA RSL Res	9.8	Region 3 BTAG	0.39	CS PSL	7
Barium	7440-39-3	1500	N	330	Invert EPA SSL	330	EPA SSL Invert	48	NOAA SQuiRT	48	Sed PSL	5500
Beryllium	7440-41-7	16	N	10	Plant ORNL	10	ORNL Plant	--	--	10	CS PSL	0.4
Cadmium	7440-43-9	7	N	0.36	Wildlife EPA SSL	0.36	EPA SSL Wildlife	0.99	Region 3 BTAG	0.36	CS PSL	39
Calcium	7440-70-2	--	--	--	--	--	--	--	--	--	--	-
Chromium	7440-47-3	0.29	C	0.4	Invert ORNL	0.29	EPA RSL Res	43.4	Region 3 BTAG	0.29	CS PSL	390
Cobalt	7440-48-4	2.3	N	13	Plant EPA SSL	2.3	EPA RSL Res	50	Region 3 BTAG	2.3	CS PSL	-
Copper	7440-50-8	310	N	28	Wildlife EPA SSL	28	EPA SSL Wildlife	31.6	Region 3 BTAG	28	CS PSL	3100
Iron	7439-89-6	5500	N	200	Invert ORNL	200	ORNL Invert	20000	Region 3 BTAG	200	CS PSL	-
Lead	7439-92-1	400	N	11	Wildlife EPA SSL	11	EPA SSL Wildlife	35.8	Region 3 BTAG	11	CS PSL	150
Magnesium	7439-95-4	--	--	--	--	--	--	--	--	--	--	-
Manganese	7439-96-5	180	N	220	Plant EPA SSL	180	EPA RSL Res	460	Region 3 BTAG	180	CS PSL	390
Mercury	7439-97-6	2.3	N	0.1	Invert ORNL	0.1	ORNL Invert	0.18	Region 3 BTAG	0.1	CS PSL	23
Nickel	7440-02-0	150	N	38	Plant EPA SSL	38	EPA SSL Plant	22.7	Region 3 BTAG	22.7	Sed PSL	1000
Potassium	7440-09-7	--	--	--	--	--	--	--	--	--	--	-
Selenium	7782-49-2	39	N	0.52	Plant EPA SSL	0.52	EPA SSL Plant	2	Region 3 BTAG	0.52	CS PSL	390
Silver	7440-22-4	39	N	4.2	Wildlife EPA SSL	4.2	EPA SSL Wildlife	1	Region 3 BTAG	1	Sed PSL	200
Sodium	7440-23-5	--	--	--	--	--	--	--	--	--	--	-
Thallium	7440-28-0	--	--	1	Plant ORNL	1	ORNL Plant	--	--	1	CS PSL	5.5
Vanadium	7440-62-2	39	N	2	Plant ORNL	2	ORNL Plant	57	NOAA SQuiRT	2	CS PSL	550
Zinc	7440-66-6	2300	N	46	Wildlife EPA SSL	46	EPA SSL Wildlife	121	Region 3 BTAG	46	CS PSL	6000
Petroleum Hydrocarbons												
GRO (C5-C12)	--	500	--	--	--	500	RIDEM TPH DEC	--	--	500	CS PSL	--
ExTPH (C9-C40)	--	500	--	--	--	500	RIDEM TPH DEC	--	--	500	CS PSL	--

Notes:

- Except for GRO and ExTPH, the value is the EPA Regions 3, 6, and 9 RSLs for Chemical Contaminants at Superfund Sites (EPA RSLs), Residential Soil value, May 2010 (USEPA, 2010). The value for GRO and ExTPH is the RIDEM Residential TPH DEC (RIDEM, 2004). The following notes apply:
 - One-tenth RSLs are presented for noncarcinogens (denoted with a "N" flag) to correspond to a target hazard quotient (HQ) of 0.1.
 - The RSLs for carcinogens (denoted with a "C" flag) correspond to an incremental lifetime cancer risk (ILCR) of 1E-6.
 - For the following carcinogenic analytes, the one-tenth non-carcinogenic value is less than the carcinogenic value; therefore, the one-tenth non-carcinogenic value is presented:
 - 1,2,4-Trichlorobenzene
 - 2,4,6-Trichlorophenol
 - Hexachlorobutadiene
 - Hexachloroethane
 - Aroclor-1254
 - The following notes are applicable for the RSL values for specific analytes:
 - cis- and trans-1,3-Dichloropropene - Value is for 1,3-dichloropropene.
 - 2-Nitrophenol - Value is for 2,4-dinitrophenol.
 - Dimethyl phthalate - Value is for diethyl phthalate.
 - Acenaphthylene - Value is for acenaphthene.
 - Benzo(g,h,i)perylene - Value is for pyrene.
 - Phenanthrene - Value is for pyrene.
 - Aroclors 1262 and 1268 - Value is for Aroclor 1260.
 - Chromium - Value is for hexavalent chromium (CAS No. 18540-29-9).
 - Mercury - Value is for mercury, inorganic salts (CAS No. not available).
 - Vanadium - Value is for vanadium and compounds (CAS No. not available).
- Selected ecological soil criterion from appropriate source indicated. Source criteria, notes, abbreviations, and references are presented in Appendix H, Table H-2.
- Selected ecological sediment criterion from appropriate source indicated. Source criteria, notes, abbreviations, and references are presented in Appendix H, Table H-3.
- The lower of the confirmatory soil and sediment PSLs is determined for residual material samples and presented in Worksheet #15 in order to allow comparison of residual material sample concentrations with detected concentrations in confirmatory soil and sediment samples. PSLs are not applicable for residual material.
- RIDEM Direct Exposure Criteria (DEC), residential soil values, February 2004 (RIDEM 2004). The hexavalent chromium value is presented for chromium. The RIDEM DEC residential soil values are presented for informational purposes only and were not used to determine the lowest criteria (project screening levels).

Abbreviations:

-- = Not available or not applicable
 BTAG = Biological Technical Assistance Group
 C = Carcinogenic
 NOAA = National Oceanic and Atmospheric Administration
 ORNL = Oak Ridge National Laboratory
 RIDEM = Rhode Island Department of Environmental Management

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 CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
 IR SITE 03/QDC OUTFALL 001
 NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND**

		A	B	C	D						
Analyte	CAS Number	EPA RSL Residential Soil ⁽¹⁾ = Test Pit Soil PSL (mg/kg)	Selected Ecological Soil Screening Level (SSL) ⁽²⁾ (mg/kg)	Selected Ecological SSL Reference ⁽²⁾	Lower of EPA RSL Residential Soil or Ecological SSL (Column A or B) = Confirmatory Soil PSL (mg/kg)	Confirmatory Soil PSL Reference	Ecological Sediment Screening Level ⁽³⁾ = Sediment PSL (mg/kg)	Sediment PSL Reference ⁽³⁾	Lower of Confirmatory Soil PSL or Sediment PSL (Column C or D) = Residual Material PSL ⁽⁴⁾ (mg/kg)	Lower of Confirmatory Soil PSL or Sediment PSL Reference ⁽⁴⁾	RIDEM DEC - Residential Soil (mg/kg) ⁽⁵⁾

CS = Confirmatory Soil
 Eco = Ecological
 ESL = Ecological Screening Level
 ExTPH = Extractable TPH
 GRO = Gasoline Range Organics
 Invert = Invertebrate
 N = Non-carcinogenic

RSL - Regional Screening Level
 SCV = Secondary Chronic Value
 SQB = Sediment Quality Benchmarks
 SQUIRT = Screening Quick Reference Tables
 SSL = Soil Screening Level
 TPH = Total Petroleum Hydrocarbons
 USEPA = United States Environmental Protection Agency

TABLE H-2
SOIL ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Soil Ecological Screening Criterion ⁽¹⁾						Selected Soil Eco Criterion	Source of Selected Soil Eco Criterion
		Plant		Invertebrate		Wildlife	USEPA Region 5 ESL		
		USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL			
Volatile Organic Compounds (mg/kg)									
1,1,1-Trichloroethane	71-55-6	NA	NA	NA	NA	NA	29.8	29.8	Region 5 ESL
1,1,2,2-Tetrachloroethane	79-34-5	NA	NA	NA	NA	NA	0.127	0.127	Region 5 ESL
1,1,2-Trichloroethane	79-00-5	NA	NA	NA	NA	NA	28.6	28.6	Region 5 ESL
1,1-Dichloroethane	75-34-3	NA	NA	NA	NA	NA	20.1	20.1	Region 5 ESL
1,1-Dichloroethene	75-35-4	NA	NA	NA	NA	NA	8.28	8.28	Region 5 ESL
1,2-Dichlorobenzene	95-50-1	NA	NA	NA	NA	NA	2.96	2.96	Region 5 ESL
1,2-Dichloroethane	107-06-2	NA	NA	NA	NA	NA	21.2	21.2	Region 5 ESL
1,2-Dichloropropane	78-87-5	NA	NA	NA	700	NA	32.7	32.7	Region 5 ESL
1,2,4-Trichlorobenzene	120-82-1	NA	NA	NA	20	NA	11.1	11.1	Region 5 ESL
1,2,4-Trimethylbenzene	95-63-6	NA	NA	NA	NA	NA	NA	NA	NA
1,3,5-Trimethylbenzene	108-67-8	NA	NA	NA	NA	NA	NA	NA	NA
1,3-Dichlorobenzene	541-73-1	NA	NA	NA	NA	NA	37.7	37.7	Region 5 ESL
1,4 Dichlorobenzene	106-46-7	NA	NA	NA	20	NA	0.546	0.546	Region 5 ESL
2-Butanone (MEK)	78-93-3	NA	NA	NA	NA	NA	89.6	89.6	Region 5 ESL
2-Hexanone	591-78-6	NA	NA	NA	NA	NA	12.6	12.6	Region 5 ESL
4-Methyl-2-pentanone (MIBK)	108-10-1	NA	NA	NA	NA	NA	443	443	Region 5 ESL
Acetone	67-64-1	NA	NA	NA	NA	NA	2.5	2.5	Region 5 ESL
Benzene	71-43-2	NA	NA	NA	NA	NA	0.255	0.255	Region 5 ESL
Bromodichloromethane	75-27-4	NA	NA	NA	NA	NA	0.54	0.54	Region 5 ESL
Bromoform	75-25-2	NA	NA	NA	NA	NA	15.9	15.9	Region 5 ESL
Bromomethane	74-83-9	NA	NA	NA	NA	NA	0.235	0.235	Region 5 ESL
Carbon Disulfide	75-15-0	NA	NA	NA	NA	NA	0.0941	0.0941	Region 5 ESL
Carbon Tetrachloride	56-23-5	NA	NA	NA	1000 ⁽⁴⁾	NA	2.98	2.98	Region 5 ESL
Chlorobenzene	108-90-7	NA	NA	NA	40	NA	13.1	13.1	Region 5 ESL
Chloroethane	75-00-3	NA	NA	NA	NA	NA	NA	NA	NA
Chloroform	67-66-3	NA	NA	NA	NA	NA	1.19	1.19	Region 5 ESL
Chloromethane	74-87-3	NA	NA	NA	NA	NA	10.4	10.4	Region 5 ESL
cis-1,2-Dichloroethene	156-59-2	NA	NA	NA	NA	NA	0.78373	0.78373	Region 5 ESL
cis-1,3-Dichloropropene	10061-01-5	NA	NA	NA	NA	NA	0.398	0.398	Region 5 ESL
Dibromochloromethane	124-48-1	NA	NA	NA	NA	NA	2.05	2.05	Region 5 ESL
Ethylbenzene	100-41-4	NA	NA	NA	NA	NA	5.16	5.16	Region 5 ESL
Methylene Chloride	75-09-2	NA	NA	NA	NA	NA	4.05	4.05	Region 5 ESL
Styrene	100-42-5	NA	300	NA	NA	NA	4.69	4.69	Region 5 ESL
Tetrachloroethene	127-18-4	NA	NA	NA	NA	NA	9.92	9.92	Region 5 ESL
Toluene	108-88-3	NA	200	NA	NA	NA	5.45	5.45	Region 5 ESL
trans-1,2-Dichloroethene	156-60-5	NA	NA	NA	NA	NA	0.784	0.784	Region 5 ESL

TABLE H-2
SOIL ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Soil Ecological Screening Criterion ⁽¹⁾						Selected Soil Eco Criterion	Source of Selected Soil Eco Criterion
		Plant		Invertebrate		Wildlife	USEPA Region 5 ESL		
		USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL			
trans-1,3-Dichloropropene	10061-02-6	NA	NA	NA	NA	NA	0.398	0.398	Region 5 ESL
Trichloroethene	79-01-6	NA	NA	NA	NA	NA	12.4	12.4	Region 5 ESL
Trichlorofluoromethane (CFC-11)	75-69-4	NA	NA	NA	NA	NA	16.4	16.4	Region 5 ESL
Vinyl Chloride	75-01-4	NA	NA	NA	NA	NA	0.646	0.646	Region 5 ESL
Xylenes (total)	1330-20-7	NA	NA	NA	NA	NA	10	10	Region 5 ESL
Semivolatile Organic Compounds (mg/kg)									
2,4,5-Trichlorophenol	95-95-4	NA	4	NA	9	NA	14.1	4	ORNL Plant
2,4,6-Trichlorophenol	88-06-2	NA	NA	NA	10	NA	9.94	9.94	Region 5 ESL
2,4-Dichlorophenol	120-83-2	NA	NA	NA	NA	NA	87.5	87.5	Region 5 ESL
2,4-Dimethylphenol	105-67-9	NA	NA	NA	NA	NA	0.01	0.01	Region 5 ESL
2,4-Dinitrophenol	51-28-5	NA	20	NA	NA	NA	0.0609	0.0609	Region 5 ESL
2,4-Dinitrotoluene	121-14-2	NA	NA	NA	NA	NA	1.28	1.28	Region 5 ESL
2,6-Dinitrotoluene	606-20-2	NA	NA	NA	NA	NA	0.0328	0.0328	Region 5 ESL
2-Chloronaphthalene	91-58-7	NA	NA	NA	NA	NA	0.0122	0.0122	Region 5 ESL
2-Chlorophenol	95-57-8	NA	NA	NA	NA	NA	0.243	0.243	Region 5 ESL
2-Methylphenol	95-48-7	NA	NA	NA	NA	NA	40.4	40.4	Region 5 ESL
2-Nitroaniline	88-74-4	NA	NA	NA	NA	NA	74.1	74.1	Region 5 ESL
2-Nitrophenol	88-75-5	NA	NA	NA	NA	NA	1.6	1.6	Region 5 ESL
3,3'-Dichlorobenzidine	91-94-1	NA	NA	NA	NA	NA	0.646	0.646	Region 5 ESL
3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	3.16	3.16	Region 5 ESL
4,6-Dinitro-2-methylphenol	534-52-1	NA	NA	NA	NA	NA	0.144	0.144	Region 5 ESL
4-Bromophenyl-phenylether	101-55-3	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	59-50-7	NA	NA	NA	NA	NA	7.95	7.95	Region 5 ESL
4-Chloroaniline	106-47-8	NA	NA	NA	NA	NA	1.1	1.1	Region 5 ESL
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	NA	NA	NA	NA	NA	NA
4-Methylphenol	106-44-5	NA	NA	NA	NA	NA	163	163	Region 5 ESL
4-Nitroaniline	100-01-6	NA	NA	NA	NA	NA	21.9	21.9	Region 5 ESL
4-Nitrophenol	100-02-7	NA	NA	NA	7	NA	5.12	5.12	Region 5 ESL
Bis(2-Chloroethoxy)methane	111-91-1	NA	NA	NA	NA	NA	0.302	0.302	Region 5 ESL
bis(2-Chloroethyl)ether	111-44-4	NA	NA	NA	NA	NA	23.7	23.7	Region 5 ESL
Bis(2-Ethylhexyl)phthalate	117-81-7	NA	NA	NA	NA	NA	0.925	0.925	Region 5 ESL
Butylbenzylphthalate	85-68-7	NA	NA	NA	NA	NA	0.239	0.239	Region 5 ESL
Carbazole	86-74-8	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	132-64-9	NA	NA	NA	NA	NA	NA	NA	NA
Diethylphthalate	84-66-2	NA	100	NA	NA	NA	24.8	24.8	Region 5 ESL
Dimethylphthalate	131-11-3	NA	NA	NA	200	NA	734	734	Region 5 ESL
Di-n-butylphthalate	84-74-2	NA	200	NA	NA	NA	0.15	0.15	Region 5 ESL

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NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Soil Ecological Screening Criterion ⁽¹⁾						Selected Soil Eco Criterion	Source of Selected Soil Eco Criterion
		Plant		Invertebrate		Wildlife	USEPA Region 5 ESL		
		USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL			
Di-n-octylphthalate	117-84-0	NA	NA		NA	NA	709	709	Region 5 ESL
Hexachlorobutadiene	87-68-3	NA	NA	NA	NA	NA	0.0398	0.0398	Region 5 ESL
Hexachlorocyclopentadiene	77-47-4	NA	10	NA	NA	NA	0.755	0.755	Region 5 ESL
Hexachloroethane	67-72-1	NA	NA	NA	NA	NA	0.596	0.596	Region 5 ESL
Isophorone	78-59-1	NA	NA	NA	NA	NA	139	139	Region 5 ESL
Nitrobenzene	98-95-3	NA	NA	NA	40	NA	1.31	1.31	Region 5 ESL
N-Nitroso-di-n-propylamine	621-64-7	NA	NA	NA	NA	NA	0.544	0.544	Region 5 ESL
N-Nitrosodiphenylamine	86-30-6	NA	NA	NA	20	NA	0.545	0.545	Region 5 ESL
Pentachlorophenol	87-86-5	5	3	31	6	2.1	0.119	2.1	EPA SSL Wildlife
Phenol	108-95-2	NA	70	NA	30	NA	120	30	ORNL Invert
Polycyclic Aromatic Hydrocarbons (mg/kg)									
2-Methylnaphthalene	91-57-6	NA	NA	29	NA	100	3.24	29	EPA SSL Invert
Acenaphthene	83-32-9	NA	20	29	NA	100	682	20	ORNL Plant
Acenaphthylene	208-96-8	NA	NA	29	NA	100	682	29	EPA SSL Invert
Anthracene	120-12-7	NA	NA	29	NA	100	1480	29	EPA SSL Invert
Benzo(a)anthracene	56-55-3	NA	NA	18	NA	1.1	5.21	1.1	EPA SSL Wildlife
Benzo(a)pyrene	50-32-8	NA	NA	18	NA	1.1	1.52	1.1	EPA SSL Wildlife
Benzo(b)fluoranthene	205-99-2	NA	NA	18	NA	1.1	59.8	1.1	EPA SSL Wildlife
Benzo(g,h,i)perylene	191-24-2	NA	NA	18	NA	1.1	119	1.1	EPA SSL Wildlife
Benzo(k)fluoranthene	207-08-9	NA	NA	18	NA	1.1	148	1.1	EPA SSL Wildlife
Chrysene	218-01-9	NA	NA	18	NA	1.1	4.73	1.1	EPA SSL Wildlife
Dibenzo(a,h)anthracene	53-70-3	NA	NA	18	NA	1.1	18.4	1.1	EPA SSL Wildlife
Fluoranthene	206-44-0	NA	NA	29	NA	100	122	29	EPA SSL Invert
Fluorene	86-73-7	NA	NA	29	30	100	122	29	EPA SSL Invert
Indeno(1,2,3-c,d)pyrene	193-39-5	NA	NA	18	NA	1.1	109	1.1	EPA SSL Wildlife
Naphthalene	91-20-3	NA	NA	29	NA	100	0.0994	29	EPA SSL Invert
Phenanthrene	85-01-8	NA	NA	29	NA	100	45.7	29	EPA SSL Invert
Pyrene	129-00-0	NA	NA	18	NA	1.1	78.5	1.1	EPA SSL Wildlife
PCBs (Aroclors) (mg/kg)									
Aroclor-1016	12674-11-2	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1221	11104-28-2	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1232	11141-16-5	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1242	53469-21-9	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1248	12672-29-6	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1254	11097-69-1	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1260	11096-82-5	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL

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NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Soil Ecological Screening Criterion ⁽¹⁾						Selected Soil Eco Criterion	Source of Selected Soil Eco Criterion
		Plant		Invertebrate		Wildlife	USEPA Region 5 ESL		
		USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL			
Aroclor-1262	37324-23-5	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Aroclor-1268	11100-14-4	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
PCBs (Total)	1336-36-3	NA	40	NA	NA	NA	0.000332⁽³⁾	0.000332	Region 5 ESL
Metals (mg/kg)									
Aluminum	7429-90-5	NA ⁽²⁾	50	NA ⁽²⁾	600 ⁽⁴⁾	NA	NA	50	ORNL Plant
Antimony	7440-36-0	NA	5	78	NA	0.27	0.142	0.27	EPA SSL Wildlife
Arsenic	7440-38-2	18	10	NA	60	43	5.7	18	EPA SSL Plant
Barium	7440-39-3	NA	500	330	3000 ⁽⁴⁾	2000	1.04	330	EPA SSL Invert
Beryllium	7440-41-7	NA	10	40	NA	21	1.06	10	ORNL Plant
Cadmium	7440-43-9	32	4	140	20	0.36	0.00222	0.36	EPA SSL Wildlife
Calcium	7440-70-2	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	7440-47-3	NA	1	NA	0.4	26	0.4	0.4	ORNL Invert
Cobalt	7440-48-4	13	20	NA	1000 ⁽⁴⁾	120	0.14	13	EPA SSL Plant
Copper	7440-50-8	70	100	80	60	28	5.4	28	EPA SSL Wildlife
Iron	7439-89-6	NA ⁽²⁾	NA	NA	200⁽⁶⁾	NA	NA	200	ORNL Invert
Lead	7439-92-1	120	50	1700	500	11	0.0537	11	EPA SSL Wildlife
Magnesium	7439-95-4	NA	NA	NA	NA	NA	NA	NA	NA
Manganese	7439-96-5	220	500	450	100 ⁽⁴⁾	4000	NA	220	EPA SSL Plant
Mercury	7439-97-6	NA	0.3	NA	0.1	NA	0.1	0.1	ORNL Invert
Nickel	7440-02-0	38	30	280	200	130	13.6	38	EPA SSL Plant
Potassium	7440-09-7	NA	NA	NA	NA	NA	NA	NA	NA
Selenium	7782-49-2	0.52	1	4.1	70	0.63	0.0276	0.52	EPA SSL Plant
Silver	7440-22-4	560	2	NA	50 ⁽⁴⁾	4.2	4.04	4.2	EPA SSL Wildlife
Sodium	7440-23-5	NA	NA	NA	NA	NA	NA	NA	NA
Thallium	7440-28-0	NA	1	NA	NA	NA	0.0569	1	ORNL Plant
Vanadium	7440-62-2	NA	2	NA	20 ⁽⁴⁾	7.8	1.59	2	ORNL Plant
Zinc	7440-66-6	160	50	120	100	46	6.62	46	EPA SSL Wildlife
Total Petroleum Hydrocarbons (TPH) (mg/kg)									
Gasoline Range Organics (C5-C12)	-	NA	NA	NA	NA	NA	NA	NA	NA
Extractable TPH (C9-C40)	-	NA	NA	NA	NA	NA	NA	NA	NA

Footnotes:

- (1) - Full reference is provided below. Shaded cells are values that are selected as the screening values for the investigation.
- (2) - Aluminum is considered a COPC only when the soil pH is less than 5.5;
Iron is not expected to be toxic to plants with a soil pH between 5 and 8.
- (3) - Total PCB is used as a surrogate
- (4) - Based on microorganisms

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SOIL ECOLOGICAL SCREENING CRITERIA
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Analyte	CAS No.	Soil Ecological Screening Criterion ⁽¹⁾						Selected Soil Eco Criterion	Source of Selected Soil Eco Criterion
		Plant		Invertebrate		Wildlife	USEPA Region 5 ESL		
		USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL	ORNL Benchmark	USEPA Eco SSL			

References:

- Plant, Invertebrate, and Wildlife USEPA Eco SSL (EPA SSL Plant, Invert, Wildlife) - There is a separate USEPA 2003, 2005, 2006, 2007, or 2008 reference for each analyte, as follows:
 - USEPA. 2003a. Ecological Soil Screening Levels for Aluminum. OSWER Directive 9285.7-60. February.
 - USEPA. 2003b. Ecological Soil Screening Levels for Iron. OSWER Directive 9285.7-69. November.
 - USEPA. 2005b. Ecological Soil Screening Levels for Antimony. OSWER Directive 9285.7-61. February.
 - USEPA. 2005c. Ecological Soil Screening Levels for Arsenic. OSWER Directive 9285.7-62. March.
 - USEPA. 2005d. Ecological Soil Screening Levels for Barium. OSWER Directive 9285.7-63. February.
 - USEPA. 2005e. Ecological Soil Screening Levels for Beryllium. OSWER Directive 9285.7-64. February.
 - USEPA. 2005f. Ecological Soil Screening Levels for Cadmium. OSWER Directive 9285.7-65. March.
 - USEPA. 2005g. Ecological Soil Screening Levels for Cobalt. OSWER Directive 9285.7-67. March.
 - USEPA. 2005h. Ecological Soil Screening Levels for Lead. OSWER Directive 9285.7-70. March.
 - USEPA. 2005i. Ecological Soil Screening Levels for Vanadium. OSWER Directive 9285.7-75. April.
 - USEPA, 2006. Ecological Soil Screening Level for Silver, Interim Final. OSWER Directive 9285.7-77. October.
 - USEPA, 2007a. Ecological Soil Screening Level for Copper, Interim Final. OSWER Directive 9285.7-68. February.
 - USEPA, 2007b. Ecological Soil Screening Level for Nickel, Interim Final. OSWER Directive 9285.7-76. March.
 - USEPA, 2007d. Ecological Soil Screening Level for Manganese, Interim Final. OSWER Directive 9285.7-71. April.
 - USEPA. 2007e. Ecological Soil Screening Levels for Pentachlorophenol. OSWER Directive 9285.7-58. April.
 - USEPA, 2007f. Ecological Soil Screening Level for PAHs, Interim Final. OSWER Directive 9285.7-78. June.
 - USEPA, 2007h. Ecological Soil Screening Level for Selenium, Interim Final. OSWER Directive 9285.7-72. November.
 - USEPA, 2007i. Ecological Soil Screening Level for Zinc, Interim Final. OSWER Directive 9285.7-73. November.
 - USEPA. 2008. Ecological Soil Screening Levels for Chromium. OSWER Directive 9285.7-66. April.
- Plant ORNL Benchmark (ORNL Plant): Efrogmson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997a. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-85/R3.
- Invertebrate ORNL Benchmark (ORNL Invert): Efrogmson, R.A., M.E. Will, and G.W. Suter II. 1997b. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-126/R2.
- USEPA Region 5 ESL (Region 5 ESL): USEPA, 2003. Ecological Screening Levels. USEPA Region 5 (<http://www.epa.gov/reg5rcra/ca/edql.htm>). August.

Acronyms and Abbreviations:

- Eco = Ecological
- ESL = Ecological Screening Level
- Invert = Invertebrate
- NA = Not available or not applicable
- ORNL = Oak Ridge National Laboratory
- PAH = Polycyclic Aromatic Hydrocarbon
- PCB = Polychlorinated Biphenyl
- SSL = Soil Screening Level
- USEPA = United States Environmental Protection Agency

TABLE H-3
SEDIMENT ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Sediment Ecological Screening Criterion ⁽¹⁾				Selected Sediment Eco Criterion	Source of Selected Sediment Eco Criterion
		USEPA Region 3 BTAG	SCV	USEPA SQB ⁽²⁾	NOAA SQUIRT Sediment Benchmark ⁽³⁾		
Volatile Organic Compounds (mg/kg)							
1,1,1-Trichloroethane	71-55-6	0.0302	0.03	0.17		0.0302	Region 3 BTAG
1,1,2,2-Tetrachloroethane	79-34-5	1.36	1.4	0.94		1.36	Region 3 BTAG
1,1,2-Trichloroethane	79-00-5	1.24	1.2	NA		1.24	Region 3 BTAG
1,1-Dichloroethane	75-34-3	NA	0.027	NA		0.027	SCV
1,1-Dichloroethene	75-35-4	0.031	0.031	NA		0.031	Region 3 BTAG
1,2-Dichlorobenzene	95-50-1	0.0165	0.33	0.34		0.0165	Region 3 BTAG
1,2-Dichloroethane	107-06-2	NA	0.25	NA		0.25	SCV
1,2-Dichloropropane	78-87-5	NA	NA	NA		NA	NA
1,2,4-Trichlorobenzene	120-82-1	2.1	9.6	9.2		2.1	Region 3 BTAG
1,2,4-Trimethylbenzene	95-63-6	NA	NA	NA		NA	NA
1,3,5-Trimethylbenzene	108-67-8	NA	NA	NA		NA	NA
1,3-Dichlorobenzene	541-73-1	4.43	1.7	1.7		4.43	Region 3 BTAG
1,4-Dichlorobenzene	106-46-7	0.599	0.34	0.35		0.599	Region 3 BTAG
2-Butanone	78-93-3	NA	0.27	NA		0.27	SCV
2-Hexanone	591-78-6	NA	0.022	NA		0.022	SCV
4-Methyl-2-Pentanone	108-10-1	NA	0.033	NA		0.033	SCV
Acetone	67-64-1	NA	0.0087	NA		0.0087	SCV
Benzene	71-43-2	NA	0.16	0.057		0.16	SCV
Bromodichloromethane	75-27-4	NA	NA	NA		NA	NA
Bromoform	75-25-2	0.654	0.65	0.65		0.654	Region 3 BTAG
Bromomethane	74-83-9	NA	NA	NA		NA	NA
Carbon Disulfide	75-15-0	0.000851	0.00085	NA		0.000851	Region 3 BTAG
Carbon Tetrachloride	56-23-5	0.0642	0.047	1.2		0.0642	Region 3 BTAG
Chlorobenzene	108-90-7	0.00842	0.41	0.82		0.00842	Region 3 BTAG
Chloroethane	75-00-3	NA	NA	NA		NA	NA
Chloroform	67-66-3	NA	0.022	NA		0.022	SCV
Chloromethane	74-87-3	NA	NA	NA		NA	NA
cis-1,2-Dichloroethene	156-59-2	NA	0.4	NA		0.4	SCV
cis-1,3-Dichloropropene	10061-01-5	NA	0.000051	NA		0.000051	SCV
Dibromochloromethane	124-48-1	NA	NA	NA		NA	NA
Ethylbenzene	100-41-4	1.1	0.089	3.6		1.1	Region 3 BTAG
Methylene Chloride	75-09-2	NA	0.37	NA		0.37	SCV
Styrene	100-42-5	0.559	NA	NA		0.559	Region 3 BTAG
Tetrachloroethene	127-18-4	0.468	0.41	0.53		0.468	Region 3 BTAG
Toluene	108-88-3	NA	0.05	0.67		0.05	SCV
trans-1,2-Dichloroethene	156-60-5	1.05	0.4	NA		1.05	Region 3 BTAG

**TABLE H-3
SEDIMENT ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND**

Analyte	CAS No.	Sediment Ecological Screening Criterion ⁽¹⁾				Selected Sediment Eco Criterion	Source of Selected Sediment Eco Criterion
		USEPA Region 3 BTAG	SCV	USEPA SQB ⁽²⁾	NOAA SQUIRT Sediment Benchmark ⁽³⁾		
trans-1,3-Dichloropropene	10061-02-6	NA	0.000051	NA		0.000051	SCV
Trichloroethene	79-01-6	0.0969	0.22	1.6		0.0969	Region 3 BTAG
Trichlorofluoromethane (CFC-11)	75-69-4	NA	NA	NA		NA	NA
Vinyl Chloride	75-01-4	NA	NA	NA		NA	NA
Xylenes (total)	1330-20-7	NA	0.16	NA		0.16	SCV
Semivolatile Organic Compounds (mg/kg)							
2,4,5-Trichlorophenol	95-95-4	NA	NA	NA	0.003⁽⁴⁾	0.003	NOAA SQUIRT
2,4,6-Trichlorophenol	88-06-2	0.213	NA	NA		0.213	Region 3 BTAG
2,4-Dichlorophenol	120-83-2	0.117	NA	NA		0.117	Region 3 BTAG
2,4-Dimethylphenol	105-67-9	0.029	NA	NA		0.029	Region 3 BTAG
2,4-Dinitrophenol	51-28-5	NA	NA	NA		NA	NA
2,4-Dinitrotoluene	121-14-2	0.0416	NA	NA		0.0416	Region 3 BTAG
2,6-Dinitrotoluene	606-20-2	NA	NA	NA		NA	NA
2-Chloronaphthalene	91-58-7	NA	NA	NA		NA	NA
2-Chlorophenol	95-57-8	0.0312	NA	NA		0.0312	Region 3 BTAG
2-Methylphenol	95-48-7	NA	0.012	NA		0.012	SCV
2-Nitroaniline	88-74-4	NA	NA	NA		NA	NA
2-Nitrophenol	88-75-5	NA	NA	NA		NA	NA
3,3'-Dichlorobenzidine	91-94-1	0.127	NA	NA		0.127	Region 3 BTAG
3-Nitroaniline	99-09-2	NA	NA	NA		NA	NA
4,6-Dinitro-2-methylphenol	534-52-1	NA	NA	NA		NA	NA
4-Bromophenyl-phenylether	101-55-3	1.23	1.2	1.3		1.23	Region 3 BTAG
4-Chloro-3-methylphenol	59-50-7	NA	NA	NA		NA	NA
4-Chloroaniline	106-47-8	NA	NA	NA		NA	NA
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	NA		NA	NA
4-Methylphenol	106-44-5	0.67	NA	NA		0.67	Region 3 BTAG
4-Nitroaniline	100-01-6	NA	NA	NA		NA	NA
4-Nitrophenol	100-02-7	NA	NA	NA		NA	NA
Bis(2-Chloroethoxy)methane	111-91-1	NA	NA	NA		NA	NA
bis(2-Chloroethyl)ether	111-44-4	NA	NA	NA		NA	NA
Bis(2-Ethylhexyl)phthalate	117-81-7	0.18	890	NA		0.18	Region 3 BTAG
Butylbenzylphthalate	85-68-7	10.9	11	11		10.9	Region 3 BTAG
Carbazole	86-74-8	NA	NA	NA		NA	NA
Dibenzofuran	132-64-9	0.415	0.42	2		0.415	Region 3 BTAG
Diethylphthalate	84-66-2	0.603	0.6	0.63		0.603	Region 3 BTAG
Dimethylphthalate	131-11-3	NA	NA	NA	0.006⁽⁴⁾	0.006	NOAA SQUIRT
Di-n-butylphthalate	84-74-2	6.47	11	11		6.47	Region 3 BTAG

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SEDIMENT ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Sediment Ecological Screening Criterion ⁽¹⁾				Selected Sediment Eco Criterion	Source of Selected Sediment Eco Criterion
		USEPA Region 3 BTAG	SCV	USEPA SQB ⁽²⁾	NOAA SQuiRT Sediment Benchmark ⁽³⁾		
Di-n-octylphthalate	117-84-0	NA	NA	NA	0.061 ⁽⁴⁾	0.061	NOAA SQuiRT
Hexachlorobutadiene	87-68-3	NA	NA	NA	0.0013 ⁽⁴⁾	0.0013	NOAA SQuiRT
Hexachlorocyclopentadiene	77-47-4	NA	NA	NA		NA	NA
Hexachloroethane	67-72-1	1.027	1	1		1.027	Region 3 BTAG
Isophorone	78-59-1	NA	NA	NA		NA	NA
Nitrobenzene	98-95-3	NA	NA	NA	0.021 ⁽⁴⁾	0.021	NOAA SQuiRT
N-Nitroso-di-n-propylamine	621-64-7	NA	NA	NA		NA	
N-Nitrosodiphenylamine	86-30-6	2.68	NA	NA		2.68	Region 3 BTAG
Pentachlorophenol	87-86-5	0.504	NA	NA		0.504	Region 3 BTAG
Phenol	108-95-2	0.42	NA	NA		0.42	Region 3 BTAG
Polycyclic Aromatic Hydrocarbons (mg/kg)							
2-Methylnaphthalene	91-57-6	0.0202	NA	NA		0.0202	Region 3 BTAG
Acenaphthene	83-32-9	0.0067	NA	NA		0.0067	Region 3 BTAG
Acenaphthylene	208-96-8	0.0059	NA	NA		0.0059	Region 3 BTAG
Anthracene	120-12-7	0.0572	0.22	NA		0.0572	Region 3 BTAG
Benzo(a)anthracene	56-55-3	0.108	0.11	NA		0.108	Region 3 BTAG
Benzo(a)pyrene	50-32-8	0.15	0.14	NA		0.15	Region 3 BTAG
Benzo(b)fluoranthene	205-99-2	NA	NA	NA	0.13 ⁽⁴⁾	0.13	NOAA SQuiRT
Benzo(g,h,i)perylene	191-24-2	0.17	NA	NA		0.17	Region 3 BTAG
Benzo(k)fluoranthene	207-08-9	0.24	NA	NA		0.24	Region 3 BTAG
Chrysene	218-01-9	0.166	NA	NA		0.166	Region 3 BTAG
Dibenzo(a,h)anthracene	53-70-3	0.033	NA	NA		0.033	Region 3 BTAG
Fluoranthene	206-44-0	0.423	NA	NA		0.423	Region 3 BTAG
Fluorene	86-73-7	0.0774	0.54	0.54		0.0774	Region 3 BTAG
Indeno(1,2,3-c,d)pyrene	193-39-5	0.017	NA	NA		0.017	Region 3 BTAG
Naphthalene	91-20-3	0.176	0.24	0.48		0.176	Region 3 BTAG
Phenanthrene	85-01-8	0.204	NA	NA		0.204	Region 3 BTAG
Pyrene	129-00-0	0.195	NA	NA		0.195	Region 3 BTAG
PCBs (Aroclors) (mg/kg)							
Aroclor-1016	12674-11-2	0.0598 ⁽⁵⁾	0.81 ⁽⁶⁾	NA		0.0598	Region 3 BTAG
Aroclor-1221	11104-28-2	0.0598 ⁽⁵⁾	0.12	NA		0.0598	Region 3 BTAG
Aroclor-1232	11141-16-5	0.0598 ⁽⁵⁾	0.6	NA		0.0598	Region 3 BTAG
Aroclor-1242	53469-21-9	0.0598 ⁽⁵⁾	0.17	NA		0.0598	Region 3 BTAG
Aroclor-1248	12672-29-6	0.0598 ⁽⁵⁾	1	NA		0.0598	Region 3 BTAG
Aroclor-1254	11097-69-1	0.0598 ⁽⁵⁾	0.81	NA		0.0598	Region 3 BTAG
Aroclor-1260	11096-82-5	0.0598 ⁽⁵⁾	4500	NA		0.0598	Region 3 BTAG

TABLE H-3
SEDIMENT ECOLOGICAL SCREENING CRITERIA
CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
IR SITE 03/QDC OUTFALL 001
NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND

Analyte	CAS No.	Sediment Ecological Screening Criterion ⁽¹⁾				Selected Sediment Eco Criterion	Source of Selected Sediment Eco Criterion
		USEPA Region 3 BTAG	SCV	USEPA SQB ⁽²⁾	NOAA SQUIRT Sediment Benchmark ⁽³⁾		
Aroclor-1262	37324-23-5	0.0598 ⁽⁵⁾	NA	NA		0.0598	Region 3 BTAG
Aroclor-1268	11100-14-4	0.0598 ⁽⁵⁾	0.81 ⁽⁶⁾	NA		0.0598	Region 3 BTAG
PCBs (Total)	1336-36-3	0.0598 ⁽⁵⁾	0.81 ⁽⁶⁾	NA		0.0598	Region 3 BTAG
Metals (mg/kg)							
Aluminum	7429-90-5	NA	NA	NA	25500	25500	NOAA SQUIRT
Antimony	7440-36-0	2	NA	NA		2	Region 3 BTAG
Arsenic	7440-38-2	9.8	NA	NA		9.8	Region 3 BTAG
Barium	7440-39-3	NA	NA	NA	48 ⁽⁴⁾	48	NOAA SQUIRT
Beryllium	7440-41-7	NA	NA	NA		NA	NA
Cadmium	7440-43-9	0.99	NA	NA		0.99	Region 3 BTAG
Calcium	7440-70-2	NA	NA	NA		NA	NA
Chromium	7440-47-3	43.4	NA	NA		43.4	Region 3 BTAG
Cobalt	7440-48-4	50	NA	NA		50	Region 3 BTAG
Copper	7440-50-8	31.6	NA	NA		31.6	Region 3 BTAG
Iron	7439-89-6	20000	NA	NA		20000	Region 3 BTAG
Lead	7439-92-1	35.8	NA	NA		35.8	Region 3 BTAG
Magnesium	7439-95-4	NA	NA	NA		NA	NA
Manganese	7439-96-5	460	NA	NA		460	Region 3 BTAG
Mercury	7439-97-6	0.18	NA	NA		0.18	Region 3 BTAG
Nickel	7440-02-0	22.7	NA	NA		22.7	Region 3 BTAG
Potassium	7440-09-7	NA	NA	NA		NA	NA
Selenium	7782-49-2	2	NA	NA		2	Region 3 BTAG
Silver	7440-22-4	1	NA	NA		1	Region 3 BTAG
Sodium	7440-23-5	NA	NA	NA		NA	NA
Thallium	7440-28-0	NA	NA	NA		NA	NA
Vanadium	7440-62-2	NA	NA	NA	57 ⁽⁴⁾	57	NOAA SQUIRT
Zinc	7440-66-6	121	NA	NA		121	Region 3 BTAG
Petroleum Hydrocarbons (mg/kg)							
GRO (C5-C12)	-	NA	NA	NA		NA	NA
EXTPH (C9-C40)	-	NA	NA	NA		NA	NA

Footnotes:

- (1) Full reference is provided below. Shaded cells are values that are selected as the screening values for the investigation.
- (2) - Based on 1% Total Organic Carbon.
- (3) - Freshwater value unless otherwise noted. Values are only presented for chemicals without other screening levels.
- (4) - Saltwater value
- (5) - Value is based on total PCBs.
- (6) - Aroclor-1254 is used as a surrogate.

**TABLE H-3
 SEDIMENT ECOLOGICAL SCREENING CRITERIA
 CONFIRMATORY SAMPLING AND DRAIN LINE INVESTIGATION
 IR SITE 03/QDC OUTFALL 001
 NCBC DAVISVILLE, NORTH KINGSTOWN, RHODE ISLAND**

Analyte	CAS No.	Sediment Ecological Screening Criterion ⁽¹⁾			Selected Sediment Eco Criterion	Source of Selected Sediment Eco Criterion
		USEPA Region 3 BTAG	SCV	USEPA SQB ⁽²⁾		

References:

- USEPA Region 3 BTAG (Region 3 BTAG): USEPA, 2006. Region 3 Freshwater Sediment Screening Benchmarks. August. □□
<http://www.epa.gov/reg3hwmd/risk/eco/btag/sbv/fwsed/screenbench.htm>
- SCV: Jones, D.S., G.W. Suter II, and R.N. Hull. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Oak Ridge National Laboratory. ES/ER/TM-95/R4. November.
- USEPA SQB (EPA SQB)USEPA, 1996. ECO Update, Ecotox Thresholds. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. Intermittent Bulletin, Volume 3, Number 2. EPA540/F-95/038. January.
- NOAA SQUIRT Sediment Benchmark (NOAA SQUIRT): Buchman, M. F., 2008. NOAA Screening Quick Reference Tables, NOAA OR&R Report 08-1, Seattle, WA, Office of Response and Restoration Division, National Oceanic and Atmospheric Administration. <http://response.restoration.noaa.gov/cpr/sediment/squirt/squirt.html>

Acronyms and Abbreviations:

- BTAG = Biological Technical Assistance Group
- Eco = Ecological
- NOAA = National Oceanic and Atmospheric Administration
- SCV = Secondary Chronic Value
- SQB = Sediment Quality Benchmarks
- SQUIRT = Screening Quick Reference Tables
- USEPA = United States Environmental Protection Agency
- GRO = Gasoline Range Organics
- ExTPH = Extractable Total Petroleum Hydrocarbons