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SAMPLING AND ANALYSIS PLAN FOR SITE ASSESSMENT SITE 103, 104, AND 105
OUTLYING FILED BRONSON NAS PENSACOLA FL
2/1/2012
TETRA TECH

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001



Rev. 1
February 2012

Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Site Assessment at Site 103 – Bronson Field Flight Line Site 104 – Bronson Field Hangars Site 105 – Bronson Field Parts Yard

**Outlying Field Bronson
Pensacola, Florida**

Contract Task Order JM51

February 2012



NAS Jacksonville
Jacksonville, Florida 32212-0030



TETRA TECH

TetraTech/TAL-11-087/3383-6.1

February 6, 2012

Project Number 112G03383

Commander
NAVFAC SE
Attention: Ms. Patty Marajh-Whittemore
Remedial Project Manager
ATTN: Ajax Street, Building 135N
P.O. Box 30A
NAS Jacksonville FL 32212-0300

Reference: CLEAN Contract Number N62470-08-D-1001
Contract Task Order JM51

Subject: Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), Site Assessment at Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, and Site 105 - Bronson Field Parts Yard, Outlying Landing Field Bronson, Pensacola, Florida

Dear Ms. Patty Marajh-Whittemore:

Tetra Tech is pleased to submit the Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for a Site Assessment at Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, and Site 105 - Bronson Field Parts Yard, Outlying Landing Field Bronson, Pensacola, Florida.

Copies of this document were also sent to NAS Pensacola, and the Florida Department of Environmental Protection. All comments submitted on this document have been resolved.

If you have any questions, please call me at (850) 385-9866, extension 1353.

Sincerely yours,

Frank Lesesne, P.G.
Project Manager

FKL/lc

Attachment (1 paper, 1 CD)

c: Greg Campbell, NAS Pensacola (2 paper; 2 CDs)
David Grabka, FDEP (1 paper, 1 CD)
John Trepanowski (cover letter only)
RDM, Tetra Tech (1 unbound, 1 CD)

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

**FINAL
SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)
FOR
SITE ASSESSMENT AT
SITE 103 - BRONSON FIELD FLIGHT LINE
SITE 104 - BRONSON FIELD HANGARS
SITE105 - BRONSON FIELD PARTS YARD**

**BRONSON FIELD
PENSACOLA, FLORIDA**

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

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

**Submitted by:
Tetra Tech, Inc.
234 Mall Boulevard
King of Prussia, Pennsylvania 19406-2954**

**CLEAN CONTRACT NUMBER N62470-08-D-1001
CONTRACT TASK ORDER JM51**

FEBRUARY 2012

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

Document Title: Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), Site Assessment at Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, Site 105 - Bronson Field Parts Yard, Bronson Field, Pensacola, Florida

Lead Organization: Naval Facilities Engineering Command Southeast

Preparer's Name and Organizational Affiliation: Thomas Deck, Tetra Tech NUS, Inc.

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Preparation Date (Day/Month/Year): July 12, 2011

Investigative Organization's Project Manager: *Frank Lesesne 20 Sept 11*
Signature/Date
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Tetra Tech NUS, Inc.

Investigative Organization's Project Quality Assurance Manager: *T.E. Johnston 9-20-11*
Signature/Date
Tom Johnston
Tetra Tech NUS, Inc.

Lead Organization's Project Manager: *PAMA 9/28/2011*
Signature/Date
Patty Whittemore
Naval Facilities Engineering Command Southeast

Lead Organization Quality Assurance Officer: BOWERS.KENNETH.A.12 30092474
Signature/Date
NAVFAC QAO/Chemist
Naval Facilities Engineering Command Atlantic

Digitially signed by BOWERS.KENNETH.A.12 30092474
DN: c=US, ou=U.S. Government, ou=DoD, ou=PTU,
ou=USM, cn=BOWERS.KENNETH.A.12 30092474
Date: 2011.09.27 17:09:23 -0400

Approval Signatures: _____
Signature/Date
David Grabka
Florida Department of Environmental Protection

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

Document Title: Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), Site Assessment at Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, Site 105 - Bronson Field Parts Yard, Bronson Field, Pensacola, Florida

Lead Organization: Naval Facilities Engineering Command Southeast

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Signature/Date
Patty Whittemore
Naval Facilities Engineering Command Southeast

Lead Organization Quality Assurance Officer:

Signature/Date
NAVFAC QAO/Chemist
Naval Facilities Engineering Command Atlantic

Approval Signatures:

 1/23/12

Signature/Date
David Grabka
Florida Department of Environmental Protection

EXECUTIVE SUMMARY

This Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) encompasses Field Sampling Plan and Quality Assurance Project Plan requirements for a Site Assessment at Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, and Site 105 - Bronson Field Parts Yard, Outlying Landing Field (OLF) Bronson, hereinafter referred to as Bronson Field, located in Pensacola, Florida. This document constitutes the planning document, addressing specific protocols for sample collection, sample handling and storage, chain-of-custody, laboratory and field analyses, data validation, and data reporting.

This UFP-SAP has been prepared by Tetra Tech, Inc. on behalf of Naval Facilities Engineering Command Southeast under the Comprehensive Long-term Environmental Action Navy Contract Number N62470-08-D-1001, Contract Task Order JM51. This UFP-SAP was generated for, and complies with, applicable United States Department of Navy, Florida Department of Environmental Protection (FDEP), and United States Environmental Protection Agency (USEPA) Region 4 requirements, regulations, guidance, and technical standards. This includes the Department of Defense (DoD), Department of Energy (DOE), and USEPA Interagency Task Force environmental requirements regarding federal facilities. To comply with DoD/DOE/USEPA requirements, this UFP-SAP is presented in the format of standard worksheets specified in the Uniform Federal Policy for Quality Assurance Plans guidance document (USEPA, 2005).

Bronson Field is located east of Perdido Bay in northwest Florida; approximately 5 miles west of Pensacola, Florida, and about 1 mile from the Alabama border (see Figure ES-1). Bronson Field consists of four abandoned airstrips and the remains of old support buildings for the airfield. Bronson Field is approximately 950 acres, the majority of which is covered by grass and forest [Navy Energy and Environmental Support Activity (NEESA), 1992].

During World War II, Bronson Field was established in 1942 as Tarklin Field to provide additional airspace for the training of Naval Pilots. The name was changed to OLF Bronson Field in 1944. Bronson Field was also used to maintain sea planes and train sea plane pilots. In the late 1950s, Bronson Field was closed as an active airfield, but the runways continue to be used for touch and go landings and for helicopter training.

At the time of the Preliminary Assessment Report completed in January 1992, all the runways were inactive. However, helicopters from Combat Support Squadron 16 were still using the area for training. Morale, Welfare, and Recreation (MWR) personnel are the only current employees at Bronson Field. MWR personnel operate the campground, conduct minor maintenance of the facility, and support recreational activities. Bronson Field is now known as Blue Angel Recreation Park (NEESA, 1992).

Site 103, Bronson Field Flight Line, contains an aircraft fuel distribution system. The system was identified during the preliminary assessment as the location of five underground storage tanks (USTs) located near Hangers 1103 and 1104. Tanks 1126-1129 were identified as 25,000-gallon capacity and Tank 1130 was 15,000-gallon capacity. The tanks were constructed of steel and contained aviation fuel. The tanks supplied aviation gasoline to the fuel line and the 56 fuel service pits that are present on the Bronson Field flight line. The fuel service pits were used to refuel various aircraft. The preliminary assessment noted that the five USTs and the refueling pits were scheduled for removal. However, the fuel lines were abandoned in place (NEESA, 1992). Tank closure documentation was not available at the time of UFP-SAP preparation but a field visit identified that the fuel service pits are still in place.

Site 104, Bronson Field Hangars, is the former location of two of the four hangars (1103 and 1104) that were used in support of the facilities mission. The hangar structures are no longer present; the date they were removed is unknown. Hangars 1103 and 1104 located adjacent to Runways 9 and 18 are approximately ½ mile from Perdido Bay. Maintenance shops, kerosene tanks, lubricant oil tanks, and waste oil tanks were located at both hangars. The preliminary assessment noted that numerous solvents, fuel oils, and other oils were used at and around the hangars. Interviews with station personnel during the preliminary assessment suggest that liquid materials spilled or placed on a concrete pad may have been washed into the grass during periods of precipitation or when the pad was washed down. Interviews estimated that approximately 1,000 pounds of waste might have been released (NEESA, 1992).

Site 105, Bronson Field Parts Yard, is currently used as storage in support of the current recreational activities at OLF Bronson. Site 105 was not identified as an area of concern in the 1992 preliminary assessment; but a historical figure from June 30, 1951 identified the area of Site 105 as containing Tank 1156, a garage, and a battery house. Tank 1156 is identified in the preliminary assessment as a 2000-gallon steel gasoline tank.

This UFP-SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the investigation activities at Sites 103, 104, and 105 as required under Chapter 62-780 of the Florida Administrative Code (F.A.C.). Protocols for sample collection, handling, and storage, chain-of-custody, laboratory and field analyses, data validation, and reporting are also addressed in this UFP-SAP. The sampling methods utilized will comply with FDEP standard operating procedures. The field sampling approach to detect and begin to delineate contamination (if detected) is to screen soil samples via hand held instruments and analyze groundwater samples by a mobile laboratory. Field screening data from soil samples will be used to bias the collection of soil samples for fixed-base laboratory analysis toward

the most contaminated locations. Groundwater mobile laboratory data will be used to select locations for permanent monitoring wells to collect groundwater characterization samples for analysis by a fixed-base laboratory.

Nominal soil and groundwater sampling locations for fixed-base laboratory analysis of soil and groundwater samples will be arranged in a grid at each site to obtain comprehensive spatial coverage of the site. The fixed-base laboratory soil and groundwater data will be compared to project-specific action levels protective of human health to determine whether investigation beyond what is described in this SAP is required under Chapter 62-780, F.A.C. The field work and sampling are scheduled to begin in the first quarter of 2012. A complete schedule is detailed in SAP Worksheet #16.

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- Appendix C: Analytical Laboratories of Florida, Inc., Standard Operating Procedures and Certification
- Appendix D: Empirical Laboratories, Inc., Standard Operating Procedures and Certification

ACRONYMS AND ABBREVIATIONS

%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RDS	Percent Relative Standard Deviation
AES	Atomic Emission Spectroscopy
ALF	Analytical Laboratories of Florida
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BNA	Base/Neutral/Acid
°C	Degrees Celsius
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
DDT	Dichlorodiphenyltrichloroethane
DFTPP	Decafluorotriphenyl-phosphine
DL	Detection Limit
DoD	Department of Defense
DOE	Department of Energy
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FDOH	Florida Department of Health
FID	Flame Ionization Detector
FL-PRO	Florida Residual Petroleum Range Organic Method

ACRONYMS AND ABBREVIATIONS (CONTINUED)

FOL	Field Operations Leader
FTMR	Field Task Modification Request
GC/ECD	Gas Chromatography Electron Capture Detector
GC/FID	Gas Chromatography/Flame Ionization Detector
GC/MS	Gas Chromatography Mass Spectrometry
GCTL	Groundwater Cleanup Target Level
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HSM	Health and Safety Manager
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDW	Investigation Derived Waste
IRP	Installation Restoration Program
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
µg/L	Microgram per Liter
MCL	Maximum Contaminant Level
mg/kg	Milligram per Kilogram
mL	Milliliter
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MWR	Morale, Welfare, and Recreation
NA	Not Applicable
NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
ND	Non-detect
NEESA	Naval Energy and Environmental Support Activity
NELAP	National Environmental Laboratory Accreditation Program
NIRIS	Naval Installation Restoration Information Solution

ACRONYMS AND ABBREVIATIONS (CONTINUED)

NTU	Nephelometric Turbidity Unit
OLF	Outlying Landing Field
PAH	Polynuclear Aromatic Hydrocarbon
PAL	Project Action Limit
PCB	Polychlorinated Biphenyl
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
PQLG	Project Quantitation Limit Goal
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
RF	Response Factor
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RT	Retention Time
SA	Site Assessment
SAP	Sampling and Analysis Plan
SAR	Site Assessment Report
SCTL	Soil Cleanup Target Level
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SPLP	Synthetic Precipitate Leaching Procedure
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TBD	To Be Determined

ACRONYMS AND ABBREVIATIONS (CONTINUED)

TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TRPH	Total Recoverable Petroleum Hydrocarbons
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

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

Site Name/Number: Outlying Landing Field (OLF) Bronson Field, Pensacola, Florida
Operable Unit: Site 103 - Bronson Field Flight Line, Site 104 - Bronson Field Hangars, and Site 105 - Bronson Field Parts Yard
Contractor Name: Tetra Tech, Inc.
Contract Number: N62470-08-D-1001
Contract Title: Comprehensive Long-term Environmental Action Navy (CLEAN)
Work Assignment Number Contract Task Order (CTO) JM51

1. This Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (United States Environmental Protection Agency [USEPA], 2005) and *Guidance for Quality Assurance Project Plans, QA/G-5, QAMS* (USEPA, 2002).

2. Identify regulatory program:

National Oil and Hazardous Substances Pollution Contingency Plan (NCP); Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), and Chapter 62-780, Florida Administrative Code (F.A.C.).

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

SCOPING SESSION	DATE
Data Quality Objective (DQO) Meeting	May 5, 2011

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

TITLE	DATE
Preliminary Assessment Report, OLF Bronson, Escambia County, Florida	February, 1992

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

Florida Department of Environmental Protection (FDEP) (lead regulatory stakeholder)
Naval Air Station (NAS) Pensacola, Florida (property owner)

7. Lead organization

Naval Facilities Engineering Command Southeast (NAVFAC SE)

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

Worksheet #13 is not applicable, but has been retained and labeled as not applicable. There are no other exclusions.

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

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Patty Whittemore	Navy Remedial Project Manager (RPM) / Manages Project Activities for the Navy	NAVFAC SE IPT, Gulf Coast Building 135 NAS Jacksonville FL 32212-0300	904-542-6202	patty.whittemore@navy.mil
Greg Campbell	Installation Restoration Program (IRP) Manager / NAS Pensacola and Bronson Field Point of Contact (POC)	NAS Pensacola Public Works Center 310 John Tower Road Pensacola, FL 32508-5000	850-452-3131 Extension 3007	gregory.campbell@navy.mil
Ken Bowers	NAVFAC Quality Assurance (QA) Officer (QAO)/ Navy Chemist	NAVFAC Atlantic 6505 Hampton Blvd Norfolk VA 23508	757-322-8341	kenneth.a.bowers@navy.mil
To Be Determined (TBD)	Head of Reference Desk (Bronson Field Administrative Record)	TBD	TBD	TBD
Bonnie Capito	Administrative Record/ Librarian	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil
David Grabka	FDEP RPM/ Provides Regulator Input	FDEP 2600 Blair Stone Road, MS 4535 Tallahassee, FL 32399-2400	850-245-8997	david.grabka@dep.state.fl.us
Frank Lesesne	Tetra Tech Project Manager (PM) / Manages Project Activities	Tetra Tech 1558 Village Square Boulevard Suite 2 Tallahassee, FL 32309	850-385-9899 Extension 1353	frank.lesesne@tetrattech.com
Amber Igoe	Field Operations Leader (FOL)/ Site Safety Officer (SSO)/ Manages Field Operations and Site Safety Issues	Tetra Tech 1558 Village Square Boulevard Suite 2 Tallahassee, FL 32309	850-385-9899 Extension 1352	amber.igoe@tetrattech.com

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Matt Soltis (Health and Safety Plan [HASP] only)	Health and Safety Manager (HSM) / Manages Corporate Health and Safety Program	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8912	matt.soltis@tetrattech.com
Tom Johnston (electronic copy only)	Quality Assurance Manager (QAM) / Manages Corporate QA Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8615	tom.johnston@tetrattech.com
Kelly Carper (electronic copy only)	Project Chemist / Provides Coordination with Laboratory	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-7273	kelly.carper@tetrattech.com
Joseph Samchuck (electronic copy only)	Tetra Tech Data Validation Manager (DVM) / Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8510	joseph.samchuck@tetrattech.com
Brian Richard (electronic copy only)	Laboratory PM / Representative for Laboratory and Analytical Issues	Empirical Laboratories, LLC (Empirical) 621 Mainstream Drive, Suite 270 Nashville, TN 37228	615-345-1115	brichard@empirlabs.com
Dale Schamp	Owner/Laboratory Director	Analytical Laboratories of Florida (ALF) 535 Riverdale Road Merritt Island, FL 32953	321-258-1355	mobilealf@cs.com

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

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

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

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

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

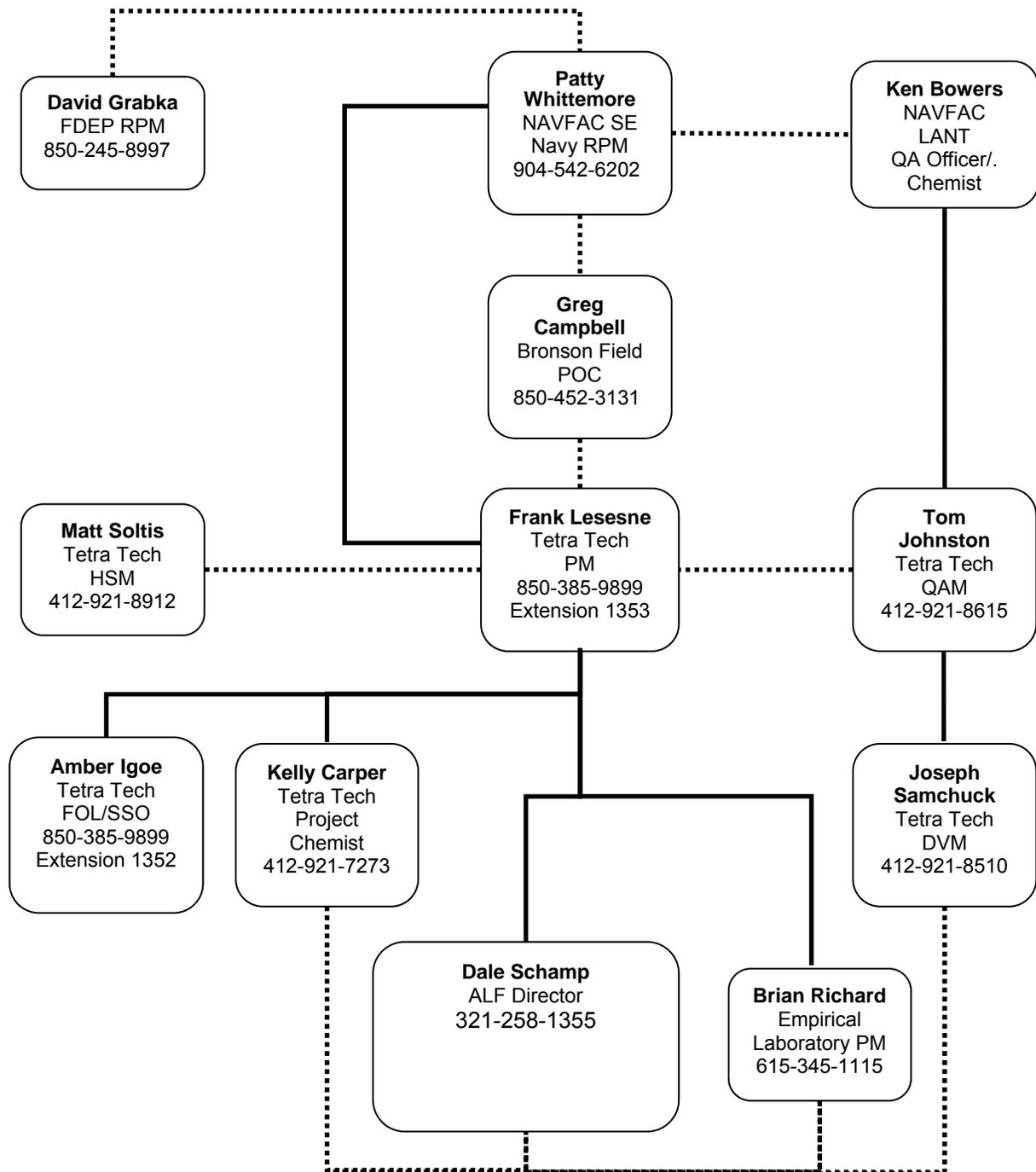
Name ¹	Organization/ Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Patty Whittemore	Navy/ RPM/ Manages Project Activities for the Navy	904-542-6202	See Worksheet #1 for signature	All	
Greg Campbell	Navy/ IRP Manager/ Bronson Field POC	850-452-3131 Extension 3007		All	
David Grabka	FDEP/ RPM/ Provides Regulator Input	850-245-8997	See Worksheet #1 for signature	All	
Tetra Tech Project Team Personnel					
Frank Lesesne	Tetra Tech/ PM / Manages Project Activities	850-385-9899 Extension 1362	See Worksheet #1 for signature	All	

Name¹	Organization/ Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Amber Igoe	Tetra Tech/ FOL/SSO Manages Field Operations and Site Safety Issues	850-385-9899 Extension 1352		All	
Tom Johnston	Tetra Tech/ QAM/ Manages NAVFAC SE Contract QA Program and Implementation	412-921-8615	See Worksheet #1 for signature	All	
Kelly Carper	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	412-921-7273		All	
Matt Soltis	Tetra Tech/ HSM/ Manages Corporate Health and Safety Program	412-921-8912	See HASP for signature	HASP	
Joseph Samchuck	Tetra Tech/ DVM/ Manages Data Validation	412-921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
Subcontractor Personnel					
Brian Richard	Empirical Laboratory PM/ Representative for Laboratory and Analytical Issues	615-345-1115		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
Dale Schamp	ALF/Laboratory director/ Representative for Laboratory and Analytical Issues	321-258-1355		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	

1 - Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.

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

Lines of Authority ————— Lines of Communication



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

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
SAP amendments	Tetra Tech FOL Tetra Tech PM Navy RPM	Amber Igoe Frank Lesesne Patty Whittemore	850-385-9899 Extension 1352 850-385-9899 Extension 1353 904-542-6202	The Tetra Tech FOL will verbally inform the Tetra Tech PM within 24 hours of realizing a need for an amendment. The Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change for review and approval. The Navy RPM will sign the letter within 5 days of receipt, if approved. The Navy RPM will notify the regulators of changes to the SAP. The Tetra Tech PM will send scope changes to the Project Team via e-mail within 1 business day.
Schedule changes	Tetra Tech PM Navy RPM Bronson Field POC	Frank Lesesne Patty Whittemore Greg Campbell	850-385-9899 Extension 1353 904-542-6202 850-452-3131 Extension 3007	The Tetra Tech PM will verbally inform the Navy RPM and the Bronson Field POC on the day that schedule change is known and document via a schedule concurrence letter within 7 days or prior to the first affected deliverable date.
Utility clearances and site access	Tetra Tech FOL	Amber Igoe	850-385-9899 Extension 1352	At least 10 days prior to commencement of field work the Tetra Tech FOL will contact the Bronson Field POC verbally to arrange for utility location marking and clearance, site access, and storage of field equipment. At least 7 days prior to commencing intrusive activities the Tetra Tech FOL will verbally contact Florida One-Call and provide the Navy IRBY Engineer and Bronson Field POC the Florida One-Call ticket number.

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL Navy RPM Tetra Tech PM	Amber Igoe Patty Whittemore Frank Lesesne	850-385-9899 Extension 1352 904-542-6202 850-385-9899 Extension 1353	The Tetra Tech FOL will inform the Tetra Tech PM verbally the day the issue is realized. The Tetra Tech PM will inform the Navy RPM of the issue (verbally or via e-mail) within 1 day of the Tetra Tech FOL's notification. Tetra Tech PM will also send a concurrence letter to the Navy RPM within 7 days, if project scope is affected. The Navy RPM will sign the letter within 5 days of receipt, if scope change is warranted. The scope change is to be implemented before further work is executed. The Tetra Tech PM will document the change via an FTMR form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form. The Tetra Tech PM will place the form in the project file, with signatures as determined by the Tetra Tech PM.
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Navy RPM Bronson Field POC	Amber Igoe Frank Lesesne Tom Johnston Patty Whittemore Greg Campbell	850-385-9899 Extension 1352 850-385-9899 Extension 1353 412-921-8615 904-542-6202 850-452-3131 Extension 3007	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform on-site personnel, subcontractor(s), the Bronson Field POC, and the identified Project Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL verbally within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
Field data quality issues	Tetra Tech FOL Tetra Tech PM	Amber Igoe Frank Lesesne	850-385-9899 Extension 1352 850-385-9899 Extension 1353	The Tetra Tech FOL will inform the Tetra Tech PM verbally or by e-mail on the same day that a field data quality issue is discovered.

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
Laboratory analytical data quality issues	Laboratory PM Laboratory Director Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Brian Richard Dale Schamp Kelly Carper Frank Lesesne Patty Whittemore	615-345-1115 321-258-1355 412-921-7273 850-385-9899 Extension 1353 904-542-6202	<p>The Laboratory PM will notify the Tetra Tech Project Chemist (verbally or via e-mail) within 1 business day of identification of a problem related to laboratory data.</p> <p>The Tetra Tech Project Chemist will notify the Tetra Tech PM and the data validation staff (verbally or via e-mail) within 1 business day.</p> <p>The Tetra Tech PM will notify the Navy RPM (verbally or via e-mail) of significant data quality issues within 1 business day of resolution.</p> <p>The Navy RPM takes corrective action that is appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the Navy Chemist.</p>

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

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

Name	Title/Role	Organizational Affiliation	Responsibilities
Patty Whittemore	RPM/ Manages project activities for the Navy	NAVFAC SE	Oversees project implementation, including scoping, data review, and evaluation.
Greg Campbell	IRP Manager/ Bronson Field POC/ Manages daily site activities related to this project	NAS Pensacola	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
David Grabka	RPM/ Provides regulatory input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP on behalf of FDEP.
Frank Lesesne	PM/ Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project.
Amber Igoe	FOL/SSO/ Manages field operations and site safety issues	Tetra Tech	Supervises, coordinates, and performs field sampling activities. As SSO, is responsible for training and monitoring site conditions. Details of these responsibilities are presented in the site-specific HASP.
Tom Johnston	QAM/ Oversees program and project QA activities	Tetra Tech	Reviews the SAP and ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Matt Soltis	HSM/ Oversees health and safety activities	Tetra Tech	Oversees Tetra Tech CLEAN Health and Safety Program.
Kelly Carper	Project Chemist/ Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.

Name	Title/Role	Organizational Affiliation	Responsibilities
Joseph Samchuck	DVM/ Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.
Brian Richard	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
Dale Schamp	Laboratory Director/ Representative for Laboratory and Analytical Issues	ALF	Coordinates analyses with laboratory analysts, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.

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

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

Each site worker performing sampling of hazardous materials will be required to have completed appropriate Hazardous Waste Operations for Emergency Response training specified in Occupational Safety and Health Administration 29 Code of Federal Regulations 1910.120(e). Safety requirements are addressed in greater detail in the site-specific Tetra Tech HASP.

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

Project Name: Site 103, Site 104, and Site 105 Projected Date(s) of Sampling: TBD Project Manager: Gerry Walker		Site Name: Site 103, Site 104, and Site 105 Site Location: Bronson Field, Pensacola, FL			
Date of Session: May 19, 2011 Scoping Session Purpose: DQO meeting to discuss additional groundwater data collection.					
Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Patty Whittemore	RPM	NAVFAC SE	904-542-6202	patty.whittemore@navy.mil	Manages Project Activities for the Navy
Ken Bowers	Navy Chemist	NAVFAC LANT	757-322-8341	kenneth.a.bowers@navy.mil	QAO for the Navy
David Grabka	RPM	FDEP	850-245-8997	david.grabka@dep.state.fl.us	Provides Regulator Input
Greg Campbell	Bronson Field POC	NAS Pensacola	850-452-3131	gregory.campbell@navy.mil	Environmental Coordinator
Frank Lesesne	PM	Tetra Tech	850-385-9866 Extension 1353	frank.lesesne@tetrattech.com	Project Management
Gerry Walker	Technical Consultant	Tetra Tech	850-385-1362	gerry.walker@tetrattech.com	Technical Support
Amber Igoe	Environmental Scientist	Tetra Tech	850-385-9866 Extension 1352	amber.igoe@tetrattech.com	Scribe
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-1300	peggy.churchill@tetrattech.com	Leads Development of DQOs
Tom Deck	DQO Support	Tetra Tech	904-730-4669 Extension 228	tom.deck@tetrattech.com	DQO support; SAP author
Kelly Carper	Project Chemist	Tetra Tech	412-921-7160	kelly.carper@tetrattech.com	Assists in completing SAP; Project Chemist

Comments/Decisions:

Site Parameter Lists identified:

Site 103 Flight Line – Target compound list (TCL) volatile organic compounds (VOCs) (tentatively identified compounds [TICs] included), TCL semivolatile organic compounds (SVOCs) (polynuclear aromatic hydrocarbons [PAHs] and TICs included), TCL polychlorinated biphenyls (PCBs), waste oil

group metals (Table C, Chapter 62-770, F.A.C.) and total recoverable petroleum hydrocarbons (TRPH). For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (e.g., PCBs) or reduced (e.g., metals) for groundwater sampling event(s).

Site 104 - TCL VOCs (TICs included), TCL SVOCs (PAHs and TICs included), TCL PCBs, target analyte list (TAL) metals and TRPH. The TAL metals will be analyzed during the first round; if any of the metals from the TAL list are non-detect (ND), then the list can be reduced. For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (e.g., PCBs) or reduced (e.g., metals) for groundwater sampling event(s).

Site 105 - TCL VOCs (TICs included), TCL SVOCs (low level PAHs and TICs included), TCL PCBs, TAL metals and TRPH. The TAL metals will be analyzed during the first round; if any of the metals from the TAL list are ND, then the list can be reduced. For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (e.g., PCBs) or reduced (e.g., metals) for groundwater sampling event(s).

Project action limits (PALs) identified during the presentation will include FDEP's criteria for leachability to groundwater.

For PCBs and metals, the following Decision Rule will be added to the UFP-SAP:

If the maximum measured target analyte concentration in soil does not exceed the PAL, then based on Project Team approval, the target analyte will not be investigated in groundwater.

The decision for the well placement will be decided after the soil and direct push technology (DPT) groundwater screening data are obtained; wells can be shifted from one Site to another if necessary and the well depths are subject to change. The Project Team will make the determination collectively after reviewing all of the analytical data.

DQO meeting minutes are included in Appendix A.

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

10.1 FACILITY BACKGROUND

OLF Bronson Field (Bronson Field) is located east of Perdido Bay in northwestern Florida, approximately 5 miles west of Pensacola, Florida, and about one mile from the Alabama border (see Figure 10-1). Bronson Field consists of four abandoned airstrips and the remains of old support buildings for the air field. Bronson Field is approximately 950 acres, the majority of which is covered by grass and forest (Navy Energy and Environmental Support Activity [NEESA], 1992).

During World War II, Bronson Field was established as Tarklin Field to provide additional airspace for the training of Naval Pilots. The name was changed to OLF Bronson Field in 1944. Bronson Field was also used to maintain sea planes and train sea plane pilots. In the late 1950's Bronson Field was closed as an active airfield, but the runways continue to be used for touch and go landings and for helicopter training.

At the time the Preliminary Assessment Report was completed in January 1992, all the runways were inactive. However, helicopters from Combat Support Squadron 16 were still using the area for training. Morale Welfare and Recreation (MWR) personnel are the only current employees at Bronson Field. MWR personnel operate the campground, conduct minor maintenance of the facility, and support recreational activities. Bronson Field is now known as Blue Angel Recreation Park (NEESA, 1992).

10.2 PHYSICAL SITE DESCRIPTION

Site 103, Bronson Field Flight Line, contains an aircraft fuel distribution system (see Figure 10-2). The system was identified during the preliminary assessment as the location of five underground storage tanks (USTs) located near Hangers 1103 and 1104. Tanks 1126 through 1129 were identified as being 25,000-gallon capacity, and Tank 1130 was identified as being 15,000-gallon capacity. The USTs were constructed of steel and contained aviation fuel. The USTs supplied aviation gasoline to the gasoline fuel line and the 56 fuel service pits that were present on the Bronson Field Flight line. The fuel service pits were used to refuel various aircraft. The preliminary assessment noted that the five USTs and the fuel service pits were scheduled for removal. However, the fuel lines were reported to have been abandoned in place (NEESA, 1992). Tank closure documentation was not available at the time of UFP-SAP preparation; but a field visit identified that the fuel service pits are still in place.

The presence of the five USTs will be confirmed during the field investigation using magnetic and ferrous metal detectors. Because the presence of the USTs at Site 103 is not definitively known, a Schonstedt MAC 51 BX Pipe and Cable Locator will be used to locate the USTs using the existing pipe line, assuming that they are still connected to the USTs. This instrument creates a magnetic field that is used to locate conductive features such as the USTs and pipeline by connecting a transmitter to one end of a metal pipe. A hand held detector picks up the enhanced magnetism of the pipeline and USTs allowing them to be easily located. An additional ferrous metal detector instrument, the Schonstedt XTpc will be used to aid in the location of the USTs. Hand auger borings and probing with a metal rod will also be conducted to confirm if the USTs are still in place.

Site 104, Bronson Field Hangars, is the former location of two of the four hangars (1103 and 1104) that were used in support of the facilities mission (see Figure 10-2). The hangar structures are no longer present; the date of removal is unknown. Hangars 1103 and 1104, which were located adjacent to Runways 9 and 18, are approximately ½ mile from Perdido Bay. Maintenance shops, kerosene tanks, lubricant oil tanks, and waste oil tanks were located at both hangars. The preliminary assessment noted that numerous solvents, fuel oils, and other oils were used at and around the hangars. Interviews with Station personnel during the preliminary assessment suggest that liquid materials spilled or placed on a concrete pad may have been washed into the grass during periods of precipitation or when the pad was washed down. Interviewees estimated that approximately 1,000 pounds of waste might have been released (NEESA, 1992).

Site 105, Bronson Field Parts Yard, is currently used as storage in support of the current recreational activities at Bronson Field (see Figure 10-2). Site 105 was not identified as an area of concern in the 1992 preliminary assessment; but a historical figure from June 30, 1951 identified the area of Site 105 as containing Structure 1156 that was used as a garage and battery house. The preliminary assessment identifies Structure 1156 as containing a 2,000-gallon steel UST that contained gasoline. Based on current aerial photographs, Structure 1156 is no longer on site. Information on structure demolition and location of the 2,000-gallon steel UST is currently not available.

The presence of USTs at Sites 104 and 105 will be confirmed during the field investigation using a ferrous metal detector. Because there is not known piping at the land surface for the USTs at Sites 104 and 105, a ferrous metal detector instrument, the Schonstedt XTpc will be used to aid in the location of the USTs. Hand auger borings and probing with a metal rod will also be conducted to confirm if the USTs are still in place.

10.3 SUMMARY OF PREVIOUS INVESTIGATIONS

In February 1992, NEESA submitted the "Preliminary Assessment Report, Naval Educational and Training Program Support Activity (NETPMSA), OLF Bronson, Escambia County, Florida" (NEESA, 1992). In this document, NEESA made the following general statements or observations regarding the Bronson Field facility:

- Between 1942 and 1950, the base used large amounts of aviation gasoline, oil products, and solvents.
- High-octane aviation gasoline (AVGAS) was used more than any other hazardous material.
- Used solvent and used oil comprised a majority of the generated hazardous waste.
- Toluene, carbon tetrachloride, and trichloroethene were a few of the solvents used.
- Eighty-five tanks were identified by NEESA during the PA (1992) at Bronson Field. All but 35 USTs at Bronson Field were contracted in 1990 to be removed by E.C. Jordon Consultants. The tanks to be removed included tanks 1126 to 1129 and tank 1130 at Site 103; tanks 1103C to 1103E and tanks 1151A to 1151C at Site 104; and tank 1156 at Site 105. The locations of tanks at Sites 103 and 104 are presented in Figure 17-1A; the location of the tank 1156 at Site 105 is unknown. Tank closure documents were not available.
- Three areas of environmental concern were discovered: two large aircraft fuel systems that were used to refuel various aircraft during 1940-1952, a hill that was used to align aircraft gun sights, and a fire fighter training area.
- Exact usage rates of fuels, oils, and solvents at Bronson Field are unknown.

Observations related specifically to Site 103, Bronson Field Flight Line, in the Preliminary Assessment Report are as follows:

- Consisted of five USTs located near Hangers 1103 and 1104. Tanks 1126-1129 were 25,000-gallon capacity, while Tank 1130 was 15,000-gallon capacity. The tanks were constructed of steel and contained aviation fuel.

- The tanks were used to supply fuel for a 5,500 foot AVGAS fuel line. The fuel line was used to transport fuel to 56 fuel service pits. The service pits were used to refuel various aircraft. The five USTs and the refueling pits were scheduled for removal. The fuel lines connected to the fuel pits were reported to have been abandoned in place.

Observations related specifically to Site 104, Bronson Field Hangars, in the Preliminary Assessment Report are as follows:

- Numerous organic solvents (halogenated and non-halogenated), organic fuels and oils were used at and around the hangars.
- Liquid materials spilled or placed on a concrete pad may have been washed into the grass during periods of precipitation or when the pad was washed down.
- Approximately 1,000 pounds of waste may have been released.

There were no observations related to Site 105, Bronson Field Parts Yard, made in the Preliminary Assessment Report outside of identifying the 2,000-gallon gasoline UST (Tank 1156) associated with the site.

10.4 CURRENT AND POTENTIAL FUTURE LAND USES

Bronson Field is currently not used as an active military facility. However, local law enforcement currently uses part of the runway for driving training. The facility is currently operated by MWR personnel and is currently used for various recreational activities (i.e., camping, sailing, and windsurfing).

There are no known future land use/development restrictions identified for Bronson Field.

10.5 CONCEPTUAL SITE MODEL

A summary of the Conceptual Site Model (CSM) based on current site conditions are shown on Figure 10-3. The text below describes the CSM.

Geology and Hydrogeology

In the southern half of Escambia County, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are separated by a thick section of relatively impermeable clay; but in the northern half, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are in contact with one

another. The upper limestone of the Floridan aquifer is separated from the lower limestone by a thick clay bed (Musgrove et. al., 1965).

The sand and gravel aquifer is composed of sand with numerous lenses and layers of clay and gravel. The formation also contains lenses of hardpan where the sand has been cemented by iron oxide minerals. This aquifer lies at the surface throughout Escambia County. Surficial sands extend from ground surface to a depth of at least 129 feet above mean sea level, below which is a 15-foot thick marine clay. Underlying the clay is more sand with numerous clay lenses (Geraghty and Miller, 1986).

Water levels in the shallow aquifer range from 10 feet to approximately 50 feet below ground surface (bgs) in the vicinity of the site. Water levels in the shallow aquifer at the subject sites are estimated to range from 10 to 15 feet bgs. The regional groundwater flow has historically been toward the Gulf of Mexico and Escambia and Perdido Rivers; however, groundwater flow can vary locally due to the effect of topography or surface water bodies. Also, the aquifer recharge is predominantly from local precipitation (Trapp, 1973).

The shallow saturated permeable beds in the sand and gravel aquifer contain groundwater under non-artesian conditions. The deeper permeable beds contain groundwater under artesian pressure, where they are confined by lenses of clay and sandy clay (NEESA, 1983).

Below the sand and gravel aquifer, the limestone layers comprise the regionally extensive Floridan aquifer, which in this area is divided into upper and lower units separated by the Bucatunna clay. The upper Floridan aquifer is an important source of water in areas east of Escambia County; however, in the Pensacola area, it is highly mineralized and not used as a water supply.

Groundwater flow on a local level is not well understood and constitutes a data gap because the local transport of contaminants is affected by local groundwater flow patterns.

Contaminant Migration Pathways and Potential Receptors

The types of chemical contaminants potentially released at Sites 103, 104, and 105 are components of organic halogenated and non-halogenated solvents, fuel oils and other oils, and metals associated with site operations. Some of the oils such as waste oils may have included PCBs and waste oil metals. Because investigations have not been conducted at Sites 103, 104 and 105, it is unknown whether organic contaminants are present in a free-phase form that currently contributes to the contamination of soil and groundwater.

Contaminants released to surface or subsurface soil may dissolve and migrate downward through the soil column with infiltrating precipitation or may migrate upwards through the soil column via volatilization.

Contaminants may have been released directly to the groundwater as a result of fuel spills or UST leaks (depending upon former UST depths) or to subsurface soil and then migrated downward as dissolved species to groundwater. Contaminants in groundwater would flow to downgradient locations. Contaminants present in surface soil can be washed to downgradient locations as suspended or dissolved species in overland flow following precipitation events. Upon collecting in wetlands or other downgradient locations, contaminants may migrate to groundwater by mechanisms similar to those governing soil contaminant transport.

The presence of chemical contamination in environmental media could pose an unacceptable health risk to humans. Human receptors potentially exposed to contamination include both current and future maintenance workers, trespassers/recreational users, future construction workers, and hypothetical future residents. However, because the current and future industrial and recreational use is not anticipated to change, maintenance workers, recreational users, and trespassers are considered the most likely receptors to contact contaminants that may be present in soils and groundwater at Sites 103, 104, and 105. The assumed exposure routes for contact with the soils for the anticipated receptors include: ingestion, dermal contact, and inhalation. The assumed exposure routes for contact with groundwater contaminants for the anticipated receptors include: ingestion, dermal contact, inhalation of volatile vapors, and vapor intrusion.

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

The following text describes the development of the Project Quality Objectives using USEPA's DQO (System Planning) Process.

11.1 PROBLEM STATEMENT

Based on Site 103, 104, and 105 operational histories and experiences at similar sites, there is a significant potential that chemical contaminants at these three sites are present in site soil and groundwater at unacceptable concentrations. The primary objective of this investigation is to determine for each of these three sites whether site-related chemicals are present in soils or groundwater at concentrations greater than those that are protective of human health and the environment and, therefore, require additional investigation in accord with Chapter 62-780, F.A.C. Because the detection of unacceptable chemical concentrations is likely, an additional objective is to gather enough data during this investigation to begin the delineation of contamination as required by Chapter 62-780, F.A.C. Data gathered from this investigation must be presented in a Site Assessment Report (SAR) and used by the Project Team to determine the path forward for each site.

11.2 INFORMATION INPUTS

In order to meet the study goals of the investigation, the physical and chemical data to be collected at Sites 103, 104, and 105 are described below:

- Organic Vapor Concentrations as Determined Using a Field Portable Flame Ionization Detector (FID) and Photoionization Detector (PID): Results of measurements made with these detectors are needed to support the selection of soil samples for fixed-base laboratory analysis.
- Field Observations: Visual and olfactory evidence (staining and odor) are needed along with FID and PID measurements for biased selection (toward locations of greatest contamination level) of subsurface soil sampling intervals.
- Mobile Laboratory Screening Data: Mobile laboratory concentrations of potential groundwater VOC contaminants must be measured to assist in selecting locations to be investigated for obtaining definitive groundwater characterization data.
- Well Stabilization and Related Data: Water table level, groundwater dissolved oxygen, conductivity, pH, temperature, turbidity, and oxidation-reduction potential data are needed for site characterization

and to determine when groundwater samples are representative of the groundwater from the aquifer being investigated.

- Fixed-base Laboratory Definitive Data: Target analyte concentrations of potential site-related contaminants in groundwater and soil are required to determine whether continued investigation is necessary and to begin delineating contamination to support future planning, if needed. Potential contaminants differ by site as follows:
 - Site 103 - VOCs, SVOCs, PCBs, waste oil metals, and TRPH in both groundwater and soils.
 - Sites 104 and 105 - VOCs, SVOCs, PCBs, TAL metals, and TRPH in both groundwater and soils.

The lists of all chemical analytical groups and individual target analytes within each group are presented in Worksheet #15. These groups include TICs and low level analyses in some cases (see Worksheet #15 for specifics). The sampling rationale and methods are presented in Worksheet #17 and Worksheet #18, and the analytical methods are presented in Worksheet #19. The selected target analytes represent those analytes that are potentially associated with historical site operations as identified in the CSM in Section 10.3.

- Background Data: Field screening data will be used to select a site specific background location for each Site. The sample location that is selected to be the site specific background will be located hydraulically upgradient of each Site and based on the field screening data will not contain site related contaminants. Background soil and groundwater samples will be collected from each site specific background location. Background soil samples will be collected from land surface to 6 inches (excluding VOCs), 6 inches to 2 feet, and, thereafter, at 2-foot intervals. The background groundwater sample will be from a shallow monitoring well that is screened across the surficial water table at is at an estimated depth of approximately 15 feet bgs.
- Groundwater Level Measurements: Synoptic groundwater levels must be measured in each monitoring well to determine the groundwater flow direction. The sampling methods are presented in Worksheet #18. Conditions under which these measurements must be made and the governing procedures are presented in Section 14.1.7.
- Horizontal and Vertical Elevations of Sampling Locations: Expressed using the Florida State Plane Coordinate system

Project Action Limits

To resolve the problem statement presented in Section 11.1, concentrations of target analytes generated by the fixed-base laboratory must be compared against PALs to resolve the problem statement. For this

investigation screening values, which are also the PALs, are listed below. For each medium, the lowest chemical-specific value (when multiple values are available) is the PAL.

Soil

- Soil Cleanup Target Levels (SCTLs) from Chapter 62-777, F.A.C., Table II (Soils) – residential direct exposure and leachability based groundwater criteria (FDEP SCTL and FDEP LEACH, respectively).
- USEPA Regions 3, 6, and 9 (June 2011) Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites - Residential soil values (EPA RSL).

Groundwater

- FDEP Groundwater Cleanup Target Levels (GCTLs) per Chapter 62-777, F.A.C., Table I (Groundwater) (FDEP GCTL).
- USEPA Regions 3, 6, and 9 (June 2011) RSLs for Chemical Contaminants at Superfund Sites-Tapwater.
- USEPA Maximum Contaminant Levels (FED MCL).

To conduct comparisons of site data to PALs, the selected fixed-base laboratory must be able to achieve Limits of Quantitation (LOQs) that are low enough to measure constituent concentrations below PALs. PALs for all media are included in Worksheet #15.

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

Several target analytes have PALs that fall between the DL and the LOQ. J-flagged data in this concentration range will be accepted to achieve project goals; however, greater scrutiny will be applied in these cases to ensure the data are representative and that decision making is not compromised by the greater uncertainty associated with true chemical concentrations in these cases. Additionally, the inability to quantify select analytes to PAL levels with confidence will be addressed in the risk assessment uncertainty analysis.

In cases where fixed-base laboratory DLs are greater than the PALs, the Project Team has agreed to replace the PAL with the laboratory LOQ for decision making purposes in accordance with the FDEP protocol (FDEP, 2004).

Worksheet #20 presents the field QC sample summary.

11.3 STUDY AREA BOUNDARIES

Two types of boundaries apply to this project – spatial and temporal. Each type of boundary is described below.

Spatial Boundaries

The populations of primary interest at each site are soil to a maximum depth of 10 feet bgs and groundwater that have been contaminated by site operations. Populations that also are of interest are soil within the shallowest 10 feet above the water table and groundwater that are not contaminated and, thus, help to bound the extent of soil and groundwater contamination in three dimensions.

The horizontal boundaries of Sites 103, 104, and 105, based on operational history and practical physical constraints, are presented on Figure 10-1. Physical constraints at Sites 103, 104 and 105 include pipe lines for the former fuel distribution system; the aircraft service pits (bowsers), and pavement for the taxiways and runways for Site 103; unknown underground utilities, paved aircraft parking areas, and the hangar floor and foundation for Site 104; unknown underground utilities and a building pad and foundation for Site 105. The spatial boundaries do not necessarily represent the extent of site-related contamination.

Site 103 includes the area in the general vicinity of the Bronson Field flight line and the aircraft fuel distribution system. Site 104 includes the area in the general vicinity of Bronson Field Hangars 1103 and 1104. Site 105 includes the area in the general vicinity of Bronson Field parts yard.

The vertical study boundary for soil at all sites extends to the groundwater (estimated to be about 10 feet below ground surface (bgs). For risk assessment, both surface and subsurface soil must be represented by the data collected during this site assessment (SA).

Note: VOCs in surface soil tend to volatilize easily; therefore, it is necessary to ensure that surface soils analyzed for VOCs are not measured in the top 6 inches of soil because volatilization from these shallowest soils would bias VOC concentrations low.

Subdividing the vertical soil column into 2-foot intervals and screening each interval to find the most contaminated interval in surface and subsurface soil is an effective approach for delineating contamination in the vertical dimension and ensuring that significant contamination is not overlooked. Surface soil consists of soil from the land surface to a depth of 2 feet, and subsurface soil consists of soil from 2 feet bgs to the water table. However, if a surficial discharge of metals or SVOCs is known or suspected (based on visual/olfactory inspection [oily residues and smells]), surface soil sampling shall be as follows: land surface to 6 inches, 6 inches to 2 feet, and, thereafter, subsurface soil at 2-foot intervals.

. If no contamination is detectable using field screening or visual and/or olfactory observations at a particular location, soil must be characterized in the shallowest 2 feet (i.e., surface soil) and in the 2-foot interval immediately above the water table smear zone. Soil contaminant concentrations in these intervals that appear to be uncontaminated is likely to be used to bound the extent of soil contamination laterally and vertically down to the water table.

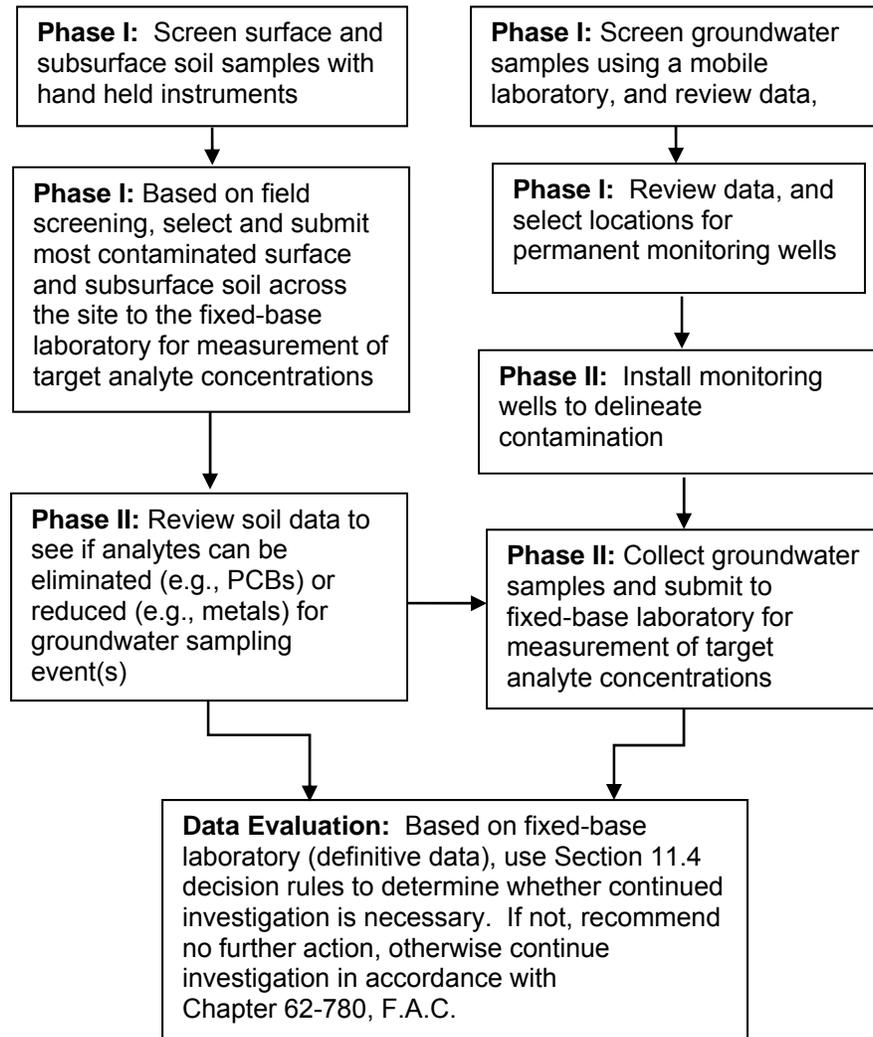
Note: To access soil underneath pavement and building, coring is necessary and must be evaluated on a site-by-site basis.

The vertical study boundary for groundwater includes the depth to the deeper aquifer zone, which is estimated to be approximately 35 feet bgs; that will be explored during this investigation. Groundwater field screening must be conducted in a screened interval in the shallow aquifer zone, estimated to be between 10 to 15 feet bgs; that is placed between the top of the water table to 5 feet below the top of the water table for analysis. During the DQO scoping session, it was decided by the Project Team that the number of shallow and deep aquifer zone monitoring well locations to collect definitive data that would be used to make regulatory decisions would be based on the groundwater field screening results (see Worksheet #9).

Temporal Boundaries

The Project Team desires to limit overall investigative costs. To support this desire, data collection for this investigation must be divided into two phases. The first phase would identify maximally contaminated soil and explore groundwater contamination sufficiently to support selection of permanent monitoring well installation locations. The second phase would be the generation of definitive soil and groundwater characterization data used to resolve the problem statement presented in Section 11.1.

The following schematic chart depicts the relationship of these phases.



11.4 ANALYTIC APPROACH

Whether further investigation is required for any of the three sites being investigated under this SAP will be determined in accordance with the following decision rules. The analytic approach developed by the Project Team (refer to Worksheet #9) for the SA includes decision rules related to characterizing the sites, delineate potential contamination, and assess potential risk.

Decision Rule #1:

Soil will be measured for VOCs, SVOCs, PCBs, TRPH and either waste oil metals or TAL metals depending on the analytical approach developed for the Site. The following decision rule will be used to determine if PCBs or metals should be measured in groundwater. If the maximum measured

target analyte PCB or metal concentration in soil does not exceed the PAL (SCTL or leachability criteria) or background sample concentration; then, based on Project Team consideration of site conditions, elect whether or not to evaluate the affected target analyte in groundwater. The tendency will be to not analyze a target analyte in groundwater if the soil concentration does not exceed the soil PAL unless site conditions and available data indicate that the investigation would benefit from analysis of the analyte in groundwater (e.g., to confirm initial indications of no groundwater contamination).

Decision Rule #2:

If the maximum measured chemical concentration does not exceed its PAL or background sample concentration in either soil or groundwater at a particular site, then exclude the chemical from further consideration and recommend Risk Management Level I, No Further Action Without Controls for that site; otherwise, retain the chemical as a contaminant of potential concern (COPC) for further assessment in accordance with Chapter 62-780, F.A.C. (see note below).

Note: Upon completion of this SA, available data may be sufficient to fully delineate contamination and conduct a risk assessment for one or more of the sites in accordance with Chapter 62-780, F.A.C. If this is the case, the Project Team may conclude during planning for further investigation, that disposition of the site can be decided without additional data collection. If this is the case, the SAR must include a full presentation of the nature and extent of contamination and the risks associated with receptor exposure to this contamination along with recommendations for disposition of the site in accordance with Chapter 62-780, F.A.C. For risk assessment purposes, if a target analyte concentration exceeds a PAL but is less than or equal to an established background concentration, the analyte will not be considered a COPC. For delineation purposes, if a background concentration for a particular analyte is greater than the PAL for that analyte, the background concentration will replace the PAL. Unless site conditions indicate otherwise, exposure point concentrations for risk assessment are to be computed using the state of Florida software called FL-UCL.

11.5 PERFORMANCE OR ACCEPTANCE CRITERIA

Simple comparisons of measured concentrations from biased sampling locations to action levels will be used for the first stages of decision making. The Project Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have PALs which are less than the laboratory method detection limits, LODs, and LOQs to ensure that contaminants are likely to have been detected, if present.

If all data have been collected as planned and no data points are missing or rejected for quality reasons, then the sampling event completeness will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether a claim of having obtained project objectives is reasonable. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented. All Project Team members will be involved in rendering the final conclusion regarding adequacy of the data.

11.6 DATA COLLECTION PLAN

The soil and groundwater sampling design, rationale, and locations are summarized in Worksheets #17 and #18. These worksheets identify the locations that are to be sampled and the analyses to be conducted.

SAP Worksheet #12 – Measurement Performance Criteria Table Field QC Samples
 (UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples ⁽¹⁾

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Trip Blank	VOCs (plus TICs)	One per cooler.	Bias/ Contamination	No target analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	S & A
Equipment Rinsate Blanks ²	All analytical groups	One per 20 samples.	Bias/ Contamination	No target analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	S & A
Field Duplicates	Organics (VOCs [plus TICs], 1,2-dibromoethane, SVOCs [including PAHs and TICs], PCBs, and TRPH)	One per 10 samples.	Precision	Soils: Relative percent difference (RPD) must be $\leq 50\%$. Waters: RPD must be $\leq 30\%$. If sample results are $< 2x$ LOQ, professional judgment is used.	S & A
	Metals (TAL and waste oil metals)	One per 10 samples.	Precision	<u>For values $\geq 5x$ LOQ</u> Soils: RPD must be $\leq 50\%$ Waters: RPD must be $\leq 30\%$. <u>For values $< 5x$ LOQ</u> Soils: Absolute difference must be $\leq 4x$ LOQ Waters: Absolute difference must be $\leq 2x$ LOQ for waters.	S & A
Cooler Temperature Indicator	All analytical groups	One per cooler.	Representativeness	Temperature must be less than or equal to 6 degrees Celsius ($^{\circ}$ C).	S

1. The Measurement Performance Criteria for laboratory QC samples are presented in Worksheet #28.
2. Equipment rinsate blanks will be collected if non-dedicated sampling equipment is used. For disposable equipment, one sample per batch of disposable equipment will be collected.

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

There are no data available for Sites 103, 104, and 15; therefore, this worksheet is not applicable.

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

14.1 SUMMARY OF PROJECT TASKS

Sampling tasks include the following:

- Mobilization and demobilization
- Health and safety training
- Utility clearance
- Soil boring/ subsurface soil sampling
- DPT Groundwater sampling
- Monitoring well installation and groundwater sampling
- Water level measurements
- Field decontamination procedure
- Investigation derived waste (IDW) management
- Documentation and records

Additional project activities include the following tasks:

- Analytical Tasks
- Data Management
- Data Review
- Project Reports

Mobilization and Demobilization

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

The fieldwork for the SA consists of two events; therefore, various selective mobilizations and demobilizations are anticipated. A brief description of the field events is as follows:

- Event 1 – DPT Investigation (Soil and Groundwater)
- Event 2 – Monitoring Well Installation and Groundwater Sampling

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

Health and Safety Training

Site-specific health and safety training per the site-specific HASP (Tetra Tech, 2011) will be provided to all Tetra Tech field crew and subcontractors as part of the site mobilization. Additionally, daily safety briefings will be conducted by the Tetra Tech FOL.

Utility Clearance

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate with Florida One-Call for utility locations. The Facility and utility companies subscribed to Sunshine State One Call will identify and mark-out utilities that may be present within the proposed soil and groundwater sampling areas. Subsurface utilities will also be cleared by the well installation subcontractor by notifying the Sunshine State One Call utility clearing service. Prior to commencing field activities the Tetra Tech FOL will:

- At least 10 days prior to commencement of field work, contact the Bronson Field POC to verbally arrange for utility location marking and clearance, and
- At least 7 days prior to commencing intrusive activities, verbally contact Florida One-Call and provide the Navy IRBY Engineer and Bronson Field POC the Florida One-Call ticket number.

See Tetra Tech Standard Operating Procedure (SOP) HS-1.0 (see Appendix B) for further information on conducting utility clearance.

Soil Boring/Subsurface Soil Sampling

Approximately 112 soil borings will be conducted using DPT at Sites 103 (44 borings), 104 (48 borings), and 105 (20 borings), and soil cores will be collected continuously from the ground surface to approximately 10 feet bgs or to the water table, whichever is encountered first.

Coring will be required to access soil underneath pavement, building pads, and foundations that are present at Sites 103, 104, and 105 prior to collecting soil samples. At Site 103, pavements include asphalt and concrete and underground pipelines that connect the former fuel distribution system. Site 104 has unknown underground utilities and paved aircraft parking areas and the hangar floor and foundation. Site 105 has unknown utilities and a building pad and foundation.

Where pavement or concrete is not present, each soil boring will be advanced initially to 4 feet using a hand auger to clear the utilities. Where pavement or concrete is present, after penetrating through the

bottom of the pavement or concrete with the DPT, each boring will be advanced initially to 4 feet using a hand auger to clear the utilities.

The soil will be described by the Tetra Tech Site Geologist and will be screened (per the methodology in Chapter 62-770 F.A.C.) for evidence of contamination with a FID/PID. Any qualitative signs of potential contamination (such as odor or staining) will be noted. Two intervals (surface and subsurface) from each boring will be submitted for off-site laboratory analysis based on field screening. The surface and subsurface soil samples from each boring shall represent the highest concentration of the field screening analysis of the soil sample intervals or visual/olfactory inspection (oily residues and smells) at the discretion of the FOL. The results of the surface and subsurface soil samples will be used to confirm the boundaries of Sites 103, 104, and 105. Soil sampling will be collected in accordance with FDEP SOP FS3000, soil logging procedures are discussed in Tetra Tech SOP GH-1.5, and the use of the PID is discussed in Tetra Tech SOP ME-12. Field SOPs are included in Appendix B.

DPT Groundwater Sampling

A mobile laboratory (ALF) will be used to screen groundwater collected from DPT soil boring location for volatile organic compounds. ALF is certified (E83934) by the Florida Department of Health (FDOH) for the matrix and analytical method that will be used during the SA. ALFs FDOH certification and SOPs are provided in Appendix C. DPT groundwater sampling methods will involve the advancement of a DPT groundwater sampling screen to a target depth. The screen is then revealed to the formation and groundwater is withdrawn via polyethylene tubing to the surface via a peristaltic pump. Groundwater samples will be collected via the straw method, placed into vials, and submitted to the mobile laboratory for analysis.

Approximately 112 groundwater samples will be collected for mobile laboratory analysis at Sites 103 (44 samples), 104 (48 samples), and 105 (20 samples). Samples will be collected according to FDEP SOP FS2200 and field screening will occur according to Tetra Tech SOP SF-1.3.

Monitoring Well Installation and Groundwater Sampling

The mobile laboratory data will be used by the Project Team to select the locations of permanent monitoring wells. Decisions will be made according to the decision tree and the decision rules presented in Worksheet #11. The intention will be to site the monitoring wells in locations that can be used to establish site-related contaminant concentration gradients so that the extent of contamination can be delineated in three dimensions and a risk assessment can be supported with the data collected from the permanent wells. Multiple 1.5-inch diameter permanent monitoring wells will be installed using DPT at Sites 103, 104, and 105 for this investigation. At Site 103, 22 shallow and 3 deep monitoring wells are planned to be installed. At Site 104, 24 shallow and 3 deep monitoring wells are planned to be installed.

At Site 105, 10 shallow and 3 deep monitoring wells are planned to be installed. All shallow monitoring wells will be installed with 10-foot screens intersecting the surficial water table to an estimated depth of approximately 15 feet bgs. All deep monitoring wells will be installed with 5-foot screens that are submerged in the surficial aquifer to an estimated depth of approximately 35 feet bgs. All shallow and deep aquifer zone monitoring wells will be installed in accordance with Tetra Tech SOP GH-2.8 (see Appendix B).

Groundwater samples will be collected from permanent monitoring wells utilizing a peristaltic pump and sterile polyethylene and medical grade silicon tubing. Purging and sampling will be conducted using the FDEP low-flow purging techniques (discharge rate of less than 1 liter per minute) with a peristaltic pump. The actual sampling depth at each monitoring well location is subject to change at the FOL's discretion based on the depth to groundwater measured in each monitoring well. All groundwater samples will be collected using the procedures specified in FDEP SOP FS 2200 (see Appendix B). Worksheets #17 and #18 specify the groundwater sampling locations and analyte groups for this investigation. Worksheet #19 specifies the analytical methods to be used.

Prior to groundwater sample collection, the monitoring wells will be purged to ensure water in each well is representative of the surrounding groundwater (formation water). Both purging and sampling operations will be conducted at a flow rate that results in a groundwater turbidity measurement of 20 Nephelometric Turbidity Units (NTUs) or less (inherent turbidity will be minimized to the greatest extent possible using low flow purging and sampling techniques; individual well conditions and local geology may preclude meeting the 20 NTU criteria, in which case it will be noted in the field logbook and sampling will proceed).

Water Level Measurements

Prior to collecting the groundwater samples, a synoptic round of electronic water level measurements will be conducted at Sites 103, 104, and 105 as part of each groundwater sampling event to provide information regarding groundwater flow patterns and hydraulic gradients. Water level measurements will be completed within the shortest time possible on the same day and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data sets. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or northern side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement event and between each monitoring well.

Field Decontamination Procedure

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

Decontamination of major equipment and sampling equipment will be in general accordance with FC 1000, Cleaning/Decontamination Procedures (FDEP, 2008), included in Appendix B.

IDW Management

It is anticipated waste materials will be generated during the field investigation. Types of IDW generated during this investigation that could be potentially contaminated include excess water or soil material collected but not placed in the laboratory supplied sample jars, sampling equipment decontamination wastewaters, and personal protective equipment (PPE) and clothing. Wastes must be disposed in such a manner that does not contribute to further environmental contamination or pose a threat to public health or safety.

Analytical results for soil and groundwater samples will be used to characterize the IDW. Additional laboratory analysis, if necessary, may be conducted for hazardous waste characterization to assess disposal options. The IDW (water or soil) will initially be placed in 55-gallon labeled, sealable steel drums. The drums will be transported to a secured area designated by the Navy. Proper disposal of these wastes will be performed by the Navy (or its designee) after the analytical results of the water or soil samples are received from the laboratory and reviewed. PPE and clothing will be wiped clean and disposed of in trash containers. Tetra Tech will send the analytical results for soil and groundwater samples to a subcontractor who will dispose of the IDW properly. The Facility is the generator and will sign (NAS Pensacola or Bronson field personnel) the waste manifest.

Tetra Tech SOP SA-7.1 located in Appendix B provides information on the handling of IDW.

Documentation and Records

Documentation of sample location coordinates, borings logs, chain-of-custody forms, samples logs, and shipping documents for samples will be recorded and filed. Preparation of electronic and hardcopies of the finalized Sites 103, 104, and 105 SA UFP-SAP will be kept on site and in the Tetra Tech CTO JM51 project file. Documentation will be conducted in accordance with FDEP SOP FD1000 (see Appendix B).

The field team will maintain a log regarding observations of site field activities while conducting field activities (test pits, DPT soil and groundwater sampling, and monitoring well installation) and collecting environmental samples for laboratory analysis. The log will follow the Field Documentation SOP provided in Appendix B.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name, site name, and site location.
- Project start date.
- Date and time of logbook entries
- Site observations including site entry and exit times
- Site sketches made on-site
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Visitor names, affiliations, arrival and departure times.
- Arrival/departure of equipment.
- Weather conditions
- Description of subcontractor activities.
- Monitoring well installation activities and operations.
- Sampling activities
- Description of borehole or monitoring well installation activities and operations.
- Sampling activities and sample log sheet references.
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Health and safety issues, including PPE.

Analytical Tasks

Fixed-base laboratory chemical analyses will be performed by Empirical, and field laboratory analyses will be performed by ALF. Each laboratory has been accredited to conduct the analyses required by this SAP. Empirical is Department of Defense (DoD) Environmental Laboratory Program (ELAP) and FDOH accredited; ALF is FDOH accredited to National ELAP (NELAP) standards. Copies of the laboratory accreditations and SOPs are located in Appendices C and D. Analyses will be performed in accordance with the analytical methods specified in Worksheet #19. The laboratory will meet the PALs as shown in Worksheet #15. The laboratory will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheet #19 and #30.

All soil results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data deliverable (EDD). This information will also be captured in the project database that will eventually be uploaded to Naval Installation Restoration Information Solution (NIRIS).

The analytical data packages provided by these laboratories will be in a contract laboratory program (CLP)-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific QC (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.).

Data Management

Data Management procedures are described in Worksheet #29.

Data Review

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

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

Project Reports

A draft SAR will be prepared with text, tables, and figures summarizing the results of all field activities and presenting all information collected. The draft SAR will be provided to all members of the Project Team for review. After resolving Project Team comments, a final SAR will be prepared and submitted to the Navy and FDEP.

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

Matrix: Groundwater
Analytical: VOCs plus TICs

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
1,1,1-TRICHLOROETHANE	71-55-6	200	FDEP GCTL	67	1	0.5	0.25
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.2	FDEP GCTL	0.067	1	0.5	0.2
1,1,2-TRICHLOROETHANE	79-00-5	5	FDEP GCTL	1.7	1	0.5	0.25
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	210000	FDEP GCTL	70000	2	1	0.5
1,1-DICHLOROETHANE	75-34-3	70	FDEP GCTL	23	1	0.5	0.25
1,1-DICHLOROETHENE	75-35-4	7	FDEP GCTL	2.3	1	0.5	0.25
1,2,3-TRICHLOROBENZENE	87-61-6	70	FDEP GCTL	23	1	0.5	0.25
1,2,4-TRICHLOROBENZENE	120-82-1	70	FDEP GCTL	23	1	0.5	0.25
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.2	FDEP GCTL	0.067	2	1	0.2
1,2-DIBROMOETHANE*	106-93-4	0.02	FDEP GCTL	0.0067	0.03	0.02	0.01
1,2-DICHLOROBENZENE	95-50-1	600	FDEP GCTL	200	1	0.5	0.25
1,2-DICHLOROETHANE	107-06-2	3	FDEP GCTL	1	1	0.5	0.25
1,2-DICHLOROPROPANE	78-87-5	5	FDEP GCTL	1.7	1	0.5	0.25
1,3-DICHLOROBENZENE	541-73-1	210	FDEP GCTL	70	1	0.5	0.25
1,4-DICHLOROBENZENE	106-46-7	75	FDEP GCTL	25	1	0.5	0.25
1,4-DIOXANE	123-91-1	3.2	FDEP GCTL	1.1	40	20	3.2
2-BUTANONE	78-93-3	4200	FDEP GCTL	1400	10	5	2.5
2-HEXANONE	591-78-6	280	FDEP GCTL	93	5	2.5	1.25
4-METHYL-2-PENTANONE	108-10-1	560	FDEP GCTL	190	5	2.5	1.25
ACETONE	67-64-1	6300	FDEP GCTL	2100	10	5	2.5
BENZENE	71-43-2	1	FDEP GCTL	0.33	1	0.5	0.25
BROMOCHLOROMETHANE	74-97-5	91	FDEP GCTL	30	1	0.5	0.25
BROMODICHLOROMETHANE	75-27-4	0.6	FDEP GCTL	0.2	1	0.5	0.25
BROMOFORM	75-25-2	4.4	FDEP GCTL	1.5	1	0.5	0.25
BROMOMETHANE	74-83-9	9.8	FDEP GCTL	3.3	2	1	0.5
CARBON DISULFIDE	75-15-0	700	FDEP GCTL	230	1	0.5	0.25
CARBON TETRACHLORIDE	56-23-5	3	FDEP GCTL	1	1	0.5	0.25
CHLOROBENZENE	108-90-7	100	FDEP GCTL	33	1	0.5	0.25
CHLORODIBROMOMETHANE	124-48-1	0.4	FDEP GCTL	0.13	1	0.5	0.25
CHLOROETHANE	75-00-3	12	FDEP GCTL	4	2	1	0.5
CHLOROFORM	67-66-3	70	FDEP GCTL	23	1	0.5	0.25
CHLOROMETHANE	74-87-3	2.7	FDEP GCTL	0.9	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP GCTL	23	1	0.5	0.25
CIS-1,3-DICHLOROPROPENE	10061-01-5	NC	NC	NC	1	0.5	0.25
CYCLOHEXANE	110-82-7	1300	Tap Water RSL	430	1	0.5	0.25

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
DICHLORODIFLUOROMETHANE	75-71-8	1400	FDEP GCTL	470	2	1	0.5
ETHYLBENZENE	100-41-4	30	FDEP GCTL	10	1	0.5	0.25
ISOPROPYLBENZENE	98-82-8	0.8	FDEP GCTL	0.27	1	0.5	0.25
M+P-XYLENES	NA	10000	FED MCL	3300	2	1	0.5
METHYL ACETATE	79-20-9	3000	FDEP GCTL	1000	2	1	0.5
METHYL CYCLOHEXANE	108-87-2	NC	NC	NC	1	0.5	0.25
METHYL TERT-BUTYL ETHER	1634-04-4	20	FDEP GCTL	6.7	1	0.5	0.25
METHYLENE CHLORIDE	75-09-2	5	FDEP GCTL	1.7	2	1	0.5
O-XYLENE	95-47-6	20	Tap Water RSL	6.7	1	0.5	0.25
STYRENE	100-42-5	100	FDEP GCTL	33	1	0.5	0.25
TETRACHLOROETHENE	127-18-4	3	FDEP GCTL	1	1	0.5	0.25
TOLUENE	108-88-3	40	FDEP GCTL	13	1	0.5	0.25
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FDEP GCTL	33	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.43	Tap Water RSL	0.14	1	0.5	0.25
TRICHLOROETHENE	79-01-6	3	FDEP GCTL	1	1	0.5	0.25
TRICHLOROFLUOROMETHANE	75-69-4	2100	FDEP GCTL	700	2	1	0.5
VINYL CHLORIDE	75-01-4	1	FDEP GCTL	0.33	1	0.5	0.25

* 1,2-Dibromoethane will be analyzed using USEPA Method SW-846 8011 separately from the other VOCs.

CAS = Chemical Abstracts Service

PQLG = Project Quantitation Limit Goal

NC = No Criteria

µg/L = microgram per liter

The PAL references for groundwater, in hierarchical order of selection, are; FDEP GCTL: Groundwater Contaminant Target Level, F.A.C. 62-777 GW-Table I (FDEP, 2005); FED MCL: USEPA Drinking Water and Health Advisories, MCL (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept this data for decision making as long as results below the LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

Shaded and Bold row indicate the PAL is less than the DL; the Partnering Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD Quality Systems Manual for Environmental Laboratories (QSM), Version 4.1.

Matrix: Groundwater

Analytical: SVOCs (including low level PAHs and TICs)

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
1,1-BIPHENYL	92-52-4	0.5	FDEP GCTL	0.17	5	2.5	0.5
1,2,4,5-TETRACHLOROBENZENE	95-94-3	2.1	FDEP GCTL	0.7	5	2.5	1.25
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	0.5	FDEP GCTL	0.17	5	2.5	0.5
2,3,4,6-TETRACHLOROPHENOL	58-90-2	210	FDEP GCTL	70	5	2.5	1.25
2,4,5-TRICHLOROPHENOL	95-95-4	1	FDEP GCTL	0.33	5	2.5	1
2,4,6-TRICHLOROPHENOL	88-06-2	3.2	FDEP GCTL	1.1	5	2.5	1.25
2,4-DICHLOROPHENOL	120-83-2	0.3	FDEP GCTL	0.1	5	2.5	0.25
2,4-DIMETHYLPHENOL	105-67-9	140	FDEP GCTL	47	20	10	5
2,4-DINITROPHENOL	51-28-5	14	FDEP GCTL	4.7	50	25	12.5
2,4-DINITROTOLUENE	121-14-2	0.05	FDEP GCTL	0.017	5	2.5	1.25
2,6-DINITROTOLUENE	606-20-2	0.05	FDEP GCTL	0.017	5	2.5	1.25
2-CHLORONAPHTHALENE	91-58-7	560	FDEP GCTL	190	5	2.5	1.25
2-CHLOROPHENOL	95-57-8	35	FDEP GCTL	12	5	2.5	1.25
2-METHYLPHENOL	95-48-7	35	FDEP GCTL	12	5	2.5	1.25
2-NITROANILINE	88-74-4	21	FDEP GCTL	7	20	10	5
2-NITROPHENOL	88-75-5	NC	NC	NC	5	2.5	1.25
3,3'-DICHLOROBENZIDINE	91-94-1	0.08	FDEP GCTL	0.027	5	2.5	1.25
3-NITROANILINE	99-09-2	1.7	FDEP GCTL	0.57	20	10	1.25
4,6-DINITRO-2-METHYLPHENOL	534-52-1	0.29	Tap Water RSL	0.097	20	10	5
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	NC	NC	5	2.5	1.25
4-CHLORO-3-METHYLPHENOL	59-50-7	63	FDEP GCTL	21	5	2.5	1.25
4-CHLOROANILINE	106-47-8	28	FDEP GCTL	9.3	5	2.5	1.25
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	NC	NC	5	2.5	1.25
4-METHYLPHENOL	106-44-5	3.5	FDEP GCTL	1.2	5	2.5	1.25
4-NITROANILINE	100-01-6	1.7	FDEP GCTL	0.57	20	10	1.25
4-NITROPHENOL	100-02-7	56	FDEP GCTL	19	20	10	5
ACETOPHENONE	98-86-2	700	FDEP GCTL	230	5	2.5	1.25
ATRAZINE	1912-24-9	3	FDEP GCTL	1	5	2.5	1.25
BENZALDEHYDE	100-52-7	700	FDEP GCTL	230	5	2.5	1.25
BIS(2-CHLOROETHOXY)METHANE	111-91-1	11	Tap Water RSL	3.7	5	2.5	1.25
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.03	FDEP GCTL	0.01	5	2.5	1.25
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	6	FDEP GCTL	2	5	2.5	1.25
BUTYL BENZYL PHTHALATE	85-68-7	140	FDEP GCTL	47	5	2.5	1.25
CAPROLACTAM	105-60-2	1800	Tap Water RSL	600	5	2.5	1.25
CARBAZOLE	86-74-8	1.8	FDEP GCTL	0.6	5	2.5	1.25
DIBENZOFURAN	132-64-9	28	FDEP GCTL	9.3	5	2.5	1.25
DIETHYL PHTHALATE	84-66-2	5600	FDEP GCTL	1900	5	2.5	1.25
DIMETHYL PHTHALATE	131-11-3	70000	FDEP GCTL	23000	5	2.5	1.25

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
DI-N-BUTYL PHTHALATE	84-74-2	700	FDEP GCTL	230	5	2.5	1.25
DI-N-OCTYL PHTHALATE	117-84-0	140	FDEP GCTL	47	5	2.5	1.25
HEXACHLOROBENZENE	118-74-1	1	FDEP GCTL	0.33	5	2.5	1
HEXACHLOROBUTADIENE	87-68-3	0.4	FDEP GCTL	0.13	5	2.5	0.25
HEXACHLOROCYCLOPENTADIENE	77-47-4	50	FDEP GCTL	17	5	2.5	1.25
HEXACHLOROETHANE	67-72-1	2.5	FDEP GCTL	0.83	5	2.5	1.25
ISOPHORONE	78-59-1	37	FDEP GCTL	12	5	2.5	1.25
NITROBENZENE	98-95-3	3.5	FDEP GCTL	1.2	5	2.5	1.25
N-NITROSODIPHENYLAMINE	86-30-6	7.1	FDEP GCTL	2.4	5	2.5	1.25
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.005	FDEP GCTL	0.0017	5	2.5	1.25
PENTACHLOROPHENOL	87-86-5	1	FDEP GCTL	0.33	2	1	0.5
PHENOL	108-95-2	10	FDEP GCTL	3.3	5	2.5	1.25
1-METHYLNAPHTHALENE	90-12-0	28	FDEP GCTL	9.3	0.2	0.1	0.05
2-METHYLNAPHTHALENE	91-57-6	28	FDEP GCTL	9.3	0.2	0.1	0.05
ACENAPHTHENE	83-32-9	20	FDEP GCTL	6.7	0.2	0.1	0.05
ACENAPHTHYLENE	208-96-8	210	FDEP GCTL	70	0.2	0.1	0.05
ANTHRACENE	120-12-7	2100	FDEP GCTL	700	0.2	0.1	0.05
BENZO(A)ANTHRACENE	56-55-3	0.05	FDEP GCTL	0.017	0.2	0.1	0.05
BENZO(A)PYRENE	50-32-8	0.2	FDEP GCTL	0.067	0.2	0.1	0.05
BENZO(B)FLUORANTHENE	205-99-2	0.05	FDEP GCTL	0.017	0.2	0.1	0.05
BENZO(G,H,I)PERYLENE	191-24-2	210	FDEP GCTL	70	0.2	0.1	0.05
BENZO(K)FLUORANTHENE	207-08-9	0.5	FDEP GCTL	0.17	0.2	0.1	0.05
CHRYSENE	218-01-9	4.8	FDEP GCTL	1.6	0.2	0.1	0.05
DIBENZO(A,H)ANTHRACENE	53-70-3	0.005	FDEP GCTL	0.0017	0.2	0.1	0.05
FLUORANTHENE	206-44-0	280	FDEP GCTL	93	0.2	0.1	0.05
FLUORENE	86-73-7	280	FDEP GCTL	93	0.2	0.1	0.05
INDENO(1,2,3-CD)PYRENE	193-39-5	0.05	FDEP GCTL	0.017	0.2	0.1	0.05
PHENANTHRENE	85-01-8	210	FDEP GCTL	70	0.2	0.1	0.05
PYRENE	129-00-0	210	FDEP GCTL	70	0.2	0.1	0.05
NAPHTHALENE	91-20-3	14	FDEP GCTL	4.7	0.2	0.1	0.05

The PAL references for groundwater, in hierarchical order of selection, are; FDEP GCTL: Groundwater Contaminant Target Level, F.A.C. 62-777 GW-Table I (FDEP, 2005); FED MCL: USEPA Drinking Water and Health Advisories, MCL (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept these data for decision making as long as results less than LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

Shaded and Bold row indicate the PAL is less than the DL; the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a “U” qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Groundwater
Analytical: PCBs

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
AROCLOR-1016	12674-11-2	0.96	Tap Water RSL	0.32	0.1	0.05	0.025
AROCLOR-1221	11104-28-2	0.0068	Tap Water RSL	0.0023	0.1	0.05	0.025
AROCLOR-1232	11141-16-5	0.0068	Tap Water RSL	0.0023	0.1	0.05	0.025
AROCLOR-1242	53469-21-9	0.034	Tap Water RSL	0.011	0.1	0.05	0.025
AROCLOR-1248	12672-29-6	0.034	Tap Water RSL	0.011	0.1	0.05	0.025
AROCLOR-1254	11097-69-1	0.034	Tap Water RSL	0.011	0.1	0.05	0.025
AROCLOR-1260	11096-82-5	0.034	Tap Water RSL	0.011	0.1	0.05	0.025

The PAL references for groundwater, in hierarchical order of selection, are; FDEP GCTL: Groundwater Contaminant Target Level, F.A.C. 62-777 GW-Table I (FDEP, 2005); FED MCL: USEPA Drinking Water and Health Advisories, MCL (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, 2011).

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Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a “U” qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Groundwater
Analytical: TAL Metals (and *Waste Oil” metals)

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
ALUMINIUM	7429-90-5	200	FDEP GCTL	67	50	25	12.5
ANTIMONY	7440-36-0	6	FDEP GCTL	2	2.5	2	1.25
ARSENIC*	7440-38-2	10	FDEP GCTL	3.3	2.5	1.5	0.75
BARIUM	7440-39-3	2000	FDEP GCTL	670	10	2.5	1.25
BERYLLIUM	7440-41-7	4	FDEP GCTL	1.3	1.25	0.5	0.25
CADMIUM*	7440-43-9	5	FDEP GCTL	1.7	1.25	0.5	0.25
CALCIUM	7440-70-2	NC	NC	NC	1250	500	250
CHROMIUM*	7440-47-3	100	FDEP GCTL	33	2.5	1	0.5
COBALT	7440-48-4	140	FDEP GCTL	47	3.125	2.5	1.25
COPPER	7440-50-8	1000	FDEP GCTL	330	2.5	2	1
IRON	7439-89-6	300	FDEP GCTL	100	25	15	7.5
LEAD*	7439-92-1	15	FDEP GCTL	5	0.75	0.75	0.375
MERCURY	7439-97-6	2	FDEP GCTL	0.67	0.2	0.2	0.08
MAGNESIUM	7439-95-4	NC	NC	NC	1250	750	250
MANGANESE	7439-96-5	50	FDEP GCTL	17	3.75	1.5	0.75
NICKEL	7440-02-0	100	FDEP GCTL	33	2.5	1.5	0.75
POTASSIUM	7440-09-7	NC	NC	NC	1250	750	250
SELENIUM	7782-49-2	50	FDEP GCTL	17	2.5	1.25	0.75
SILVER	7440-22-4	100	FDEP GCTL	33	2.5	0.5	0.25
SODIUM	7440-23-5	160000	FDEP GCTL	53000	1250	750	250
THALLIUM	7440-28-0	2	FDEP GCTL	0.67	2.0	1	0.75
VANADIUM	7440-62-2	49	FDEP GCTL	16	3.125	2.5	1.25
ZINC	7440-66-6	5000	FDEP GCTL	1700	5.0	2.5	1.25

* F.A.C. 62-770 - Waste Oil Metals

The PAL references for groundwater, in hierarchical order of selection, are; FDEP GCTL: Groundwater Contaminant Target Level, F.A.C. 62-777 GW-Table I (FDEP, 2005); FED MCL: USEPA Drinking Water and Health Advisories, MCL (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept these data for decision making as long as results less than LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

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Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a “U” qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Groundwater

Analytical: TRPH (by Florida Residual Petroleum Range Organic Method [FL-PRO])

Analyte	CAS Number	PAL (µg/L)	PAL Reference	PQLGs (µg/L)	Empirical LOQ (µg/L)	Empirical LOD (µg/L)	Empirical DL (µg/L)
TRPH	NC	5000	FDEP GCTL	1700	170	170	170

FDEP GCTL = FDEP Contaminant Target Level (CTL) 62-777 GW-Table I (FDEP)

The PAL references for groundwater, in hierarchical order of selection, are; FDEP GCTL: Groundwater Contaminant Target Level, F.A.C. 62-777 GW-Table I (FDEP, 2005); FED MCL: USEPA Drinking Water and Health Advisories, MCL (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept these data for decision making as long as results less than LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

Shaded and Bold row indicate the PAL is less than the DL; the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Soil
Analytical: VOCs plus TICs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
1,1,1-TRICHLOROETHANE	71-55-6	1.9	FDEP LEACH	0.63	0.005	0.0025	0.00125
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.001	FDEP LEACH	0.00033	0.005	0.0025	0.001
1,1,2-TRICHLOROETHANE	79-00-5	0.03	FDEP LEACH	0.01	0.005	0.0025	0.00125
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	11000	FDEP LEACH	3700	0.01	0.005	0.0025
1,1-DICHLOROETHANE	75-34-3	0.4	FDEP LEACH	0.13	0.005	0.0025	0.00125
1,1-DICHLOROETHENE	75-35-4	0.06	FDEP LEACH	0.02	0.005	0.0025	0.00125
1,2,3-TRICHLOROBENZENE	87-61-6	4.6	FDEP LEACH	1.5	0.005	0.0025	0.00125
1,2,4-TRICHLOROBENZENE	120-82-1	5.3	FDEP LEACH	1.8	0.005	0.0025	0.00125
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.001	FDEP LEACH	0.00033	0.01	0.005	0.0025
1,2-DIBROMOETHANE	106-93-4	0.0001	FDEP LEACH	0.000033	0.005	0.0025	0.00125
1,2-DICHLOROBENZENE	95-50-1	17	FDEP LEACH	5.7	0.005	0.0025	0.00125
1,2-DICHLOROETHANE	107-06-2	0.01	FDEP LEACH	0.0033	0.005	0.0025	0.00125
1,2-DICHLOROPROPANE	78-87-5	0.03	FDEP LEACH	0.01	0.005	0.0025	0.00125
1,3-DICHLOROBENZENE	541-73-1	7	FDEP LEACH	2.3	0.005	0.0025	0.00125
1,4-DICHLOROBENZENE	106-46-7	2.2	FDEP LEACH	0.73	0.005	0.0025	0.00125
1,4-DIOXANE	123-91-1	0.01	FDEP LEACH	0.0033	0.2	0.1	0.01
2-BUTANONE	78-93-3	17	FDEP LEACH	5.7	0.01	0.005	0.0025
2-HEXANONE	591-78-6	1.4	FDEP LEACH	0.47	0.005	0.0025	0.00125
4-METHYL-2-PENTANONE	108-10-1	2.6	FDEP LEACH	0.87	0.005	0.0025	0.00125
ACETONE	67-64-1	25	FDEP LEACH	8.3	0.02	0.01	0.005
BENZENE	71-43-2	0.007	FDEP LEACH	0.0023	0.005	0.0025	0.00125
BROMOCHLOROMETHANE	74-97-5	0.6	FDEP LEACH	0.2	0.005	0.0025	0.00125
BROMODICHLOROMETHANE	75-27-4	0.004	FDEP LEACH	0.0013	0.005	0.0025	0.00125
BROMOFORM	75-25-2	0.03	FDEP LEACH	0.01	0.005	0.0025	0.00125
BROMOMETHANE	74-83-9	0.05	FDEP LEACH	0.017	0.01	0.005	0.0025
CARBON DISULFIDE	75-15-0	5.6	FDEP LEACH	1.9	0.005	0.0025	0.00125
CARBON TETRACHLORIDE	56-23-5	0.04	FDEP LEACH	0.013	0.005	0.0025	0.00125
CHLOROBENZENE	108-90-7	1.3	FDEP LEACH	0.43	0.005	0.0025	0.00125
CHLORODIBROMOMETHANE	124-48-1	0.003	FDEP LEACH	0.001	0.005	0.0025	0.00125
CHLOROETHANE	75-00-3	0.06	FDEP LEACH	0.02	0.01	0.005	0.0025
CHLOROFORM	67-66-3	0.4	FDEP LEACH	0.13	0.005	0.0025	0.00125
CHLOROMETHANE	74-87-3	0.01	FDEP LEACH	0.0033	0.01	0.005	0.0025
CIS-1,2-DICHLOROETHENE	156-59-2	0.4	FDEP LEACH	0.13	0.005	0.0025	0.00125
CIS-1,3-DICHLOROPROPENE	10061-01-5	1.7	EPA RSL	0.57	0.005	0.0025	0.00125
CYCLOHEXANE	110-82-7	700	EPA RSL	230	0.005	0.0025	0.00125
DICHLORODIFLUOROMETHANE	75-71-8	44	FDEP LEACH	15	0.01	0.005	0.0025
ETHYLBENZENE	100-41-4	0.6	FDEP LEACH	0.2	0.005	0.0025	0.00125
ISOPROPYLBENZENE	98-82-8	0.2	FDEP LEACH	0.067	0.005	0.0025	0.00125

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
M+P-XYLENES	NA	NC	NC	NC	0.01	0.005	0.0025
METHYL ACETATE	79-20-9	16	FDEP LEACH	5.3	0.01	0.005	0.0025
METHYL CYCLOHEXANE	108-87-2	NC	NC	NC	0.005	0.0025	0.00125
METHYL TERT-BUTYL ETHER	1634-04-4	0.09	FDEP LEACH	0.03	0.005	0.0025	0.00125
METHYLENE CHLORIDE	75-09-2	0.02	FDEP LEACH	0.0067	0.01	0.005	0.0025
O-XYLENE	95-47-6	69	EPA RSL	23	0.005	0.0025	0.00125
STYRENE	100-42-5	3.6	FDEP LEACH	1.2	0.005	0.0025	0.00125
TETRACHLOROETHENE	127-18-4	0.03	FDEP LEACH	0.01	0.005	0.0025	0.00125
TOLUENE	108-88-3	0.5	FDEP LEACH	0.17	0.005	0.0025	0.00125
TRANS-1,2-DICHLOROETHENE	156-60-5	0.7	FDEP LEACH	0.23	0.005	0.0025	0.00125
TRANS-1,3-DICHLOROPROPENE	10061-02-6	1.7	EPA RSL	0.57	0.005	0.0025	0.00125
TRICHLOROETHENE	79-01-6	0.03	FDEP LEACH	0.01	0.005	0.0025	0.00125
TRICHLOROFLUOROMETHANE	75-69-4	33	FDEP LEACH	11	0.01	0.005	0.0025
VINYL CHLORIDE	75-01-4	0.007	FDEP LEACH	0.0023	0.005	0.0025	0.00125

mg/kg = milligram per kilogram

The PAL references for soil, in hierarchical order of selection, are; FDEP SCTL: Soil Contaminant Target Level, F.A.C. 62-777 Residential Soil-Direct Table II and Leachability Based Groundwater Criteria (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, 2011).

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Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Soil

Analytical: SVOCs (including low level PAHs and TICs)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
1,1-BIPHENYL	92-52-4	0.2	FDEP LEACH	0.067	0.333	0.167	0.0833
1,2,4,5-TETRACHLOROBENZENE	95-94-3	0.5	FDEP LEACH	0.17	0.333	0.167	0.0833
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	0.009	FDEP LEACH	0.003	0.333	0.167	0.0833
2,3,4,6-TETRACHLOROPHENOL	58-90-2	3.2	FDEP LEACH	1.1	0.333	0.167	0.0833
2,4,5-TRICHLOROPHENOL	95-95-4	0.07	FDEP LEACH	0.023	0.333	0.167	0.0417
2,4,6-TRICHLOROPHENOL	88-06-2	0.06	FDEP LEACH	0.02	0.333	0.167	0.0417
2,4-DICHLOROPHENOL	120-83-2	0.003	FDEP LEACH	0.001	0.333	0.167	0.0833
2,4-DIMETHYLPHENOL	105-67-9	1.7	FDEP LEACH	0.57	1.33	0.667	0.333
2,4-DINITROPHENOL	51-28-5	0.06	FDEP LEACH	0.02	3.33	1.67	0.833
2,4-DINITROTOLUENE	121-14-2	0.0004	FDEP LEACH	0.00013	0.333	0.167	0.0833
2,6-DINITROTOLUENE	606-20-2	0.0004	FDEP LEACH	0.00013	0.333	0.167	0.0833
2-CHLORONAPHTHALENE	91-58-7	260	FDEP LEACH	87	0.333	0.167	0.0833
2-CHLOROPHENOL	95-57-8	0.7	FDEP LEACH	0.23	0.333	0.167	0.0833
2-METHYLPHENOL	95-48-7	0.3	FDEP LEACH	0.1	0.333	0.167	0.0833
2-NITROANILINE	88-74-4	0.1	FDEP LEACH	0.033	1.33	0.667	0.1
2-NITROPHENOL	88-75-5	NC	NC	NC	0.333	0.167	0.0833
3,3'-DICHLOROBENZIDINE	91-94-1	0.003	FDEP LEACH	0.001	0.333	0.167	0.0833
3-NITROANILINE	99-09-2	0.01	FDEP LEACH	0.0033	1.33	0.667	0.333
4,6-DINITRO-2-METHYLPHENOL	534-52-1	0.4	FDEP LEACH	0.13	3.33	1.67	0.334
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	NC	NC	0.333	0.167	0.0833
4-CHLORO-3-METHYLPHENOL	59-50-7	0.4	FDEP LEACH	0.13	0.333	0.167	0.0833
4-CHLOROANILINE	106-47-8	0.2	FDEP LEACH	0.067	0.333	0.167	0.0833
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	NC	NC	0.333	0.167	0.0833
4-METHYLPHENOL	106-44-5	0.03	FDEP LEACH	0.01	0.333	0.167	0.03
4-NITROANILINE	100-01-6	0.008	FDEP LEACH	0.0027	1.33	0.667	0.333
4-NITROPHENOL	100-02-7	0.3	FDEP LEACH	0.1	1.33	0.667	0.222
ACETOPHENONE	98-86-2	3.9	FDEP LEACH	1.3	0.333	0.167	0.0833
ATRAZINE	1912-24-9	0.06	FDEP LEACH	0.02	0.333	0.167	0.0417
BENZALDEHYDE	100-52-7	4.8	FDEP LEACH	1.6	0.333	0.167	0.0833
BIS(2-CHLOROETHOXY)METHANE	111-91-1	63	FDEP LEACH	21	0.333	0.167	0.0833
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.0001	FDEP LEACH	0.000033	0.333	0.167	0.0833
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	72	FDEP SCTL	24	0.333	0.167	0.0833

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
BUTYL BENZYL PHTHALATE	85-68-7	310	FDEP LEACH	100	0.333	0.167	0.0833
CAPROLACTAM	105-60-2	3100	EPA RSL	1000	0.333	0.167	0.0833
CARBAZOLE	86-74-8	0.2	FDEP LEACH	0.067	0.333	0.167	0.0833
DIBENZOFURAN	132-64-9	15	FDEP LEACH	5	0.333	0.167	0.0833
DIETHYL PHTHALATE	84-66-2	86	FDEP LEACH	29	0.333	0.167	0.0833
DIMETHYL PHTHALATE	131-11-3	380	FDEP LEACH	130	0.333	0.167	0.0833
DI-N-BUTYL PHTHALATE	84-74-2	47	FDEP LEACH	16	0.333	0.167	0.0833
DI-N-OCTYL PHTHALATE	117-84-0	1700	FDEP SCTL	570	0.333	0.167	0.0833
HEXACHLOROBENZENE	118-74-1	0.4	FDEP SCTL	0.13	0.333	0.167	0.0833
HEXACHLOROBUTADIENE	87-68-3	1	FDEP LEACH	0.33	0.333	0.167	0.0833
HEXACHLOROCYCLOPENTADIENE	77-47-4	9.5	FDEP SCTL	3.2	0.333	0.167	0.0833
HEXACHLOROETHANE	67-72-1	0.2	FDEP LEACH	0.067	0.333	0.167	0.0833
ISOPHORONE	78-59-1	0.2	FDEP LEACH	0.067	0.333	0.167	0.0833
NITROBENZENE	98-95-3	0.02	FDEP LEACH	0.0067	0.333	0.167	0.0833
N-NITROSODIPHENYLAMINE	86-30-6	0.4	FDEP LEACH	0.13	0.333	0.167	0.0833
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.00005	FDEP LEACH	0.000017	0.333	0.167	0.0833
PENTACHLOROPHENOL	87-86-5	0.03	FDEP LEACH	0.01	1.33	0.667	0.03
PHENOL	108-95-2	0.05	FDEP LEACH	0.017	0.333	0.167	0.0417
1-METHYLNAPHTHALENE	90-12-0	3.1	FDEP LEACH	1	0.00667	0.00333	0.00167
2-METHYLNAPHTHALENE	91-57-6	8.5	FDEP LEACH	2.8	0.00667	0.00333	0.00167
ACENAPHTHENE	83-32-9	2.1	FDEP LEACH	0.7	0.00667	0.00333	0.00167
ACENAPHTHYLENE	208-96-8	27	FDEP LEACH	9	0.00667	0.00333	0.00167
ANTHRACENE	120-12-7	2500	FDEP LEACH	830	0.00667	0.00333	0.00167
BENZO(A)ANTHRACENE	56-55-3	0.15	FDEP SCTL	0.05	0.00667	0.00333	0.00167
BENZO(A)PYRENE	50-32-8	0.1	FDEP SCTL	0.033	0.00667	0.00333	0.00167
BENZO(B)FLUORANTHENE	205-99-2	0.15	FDEP SCTL	0.05	0.00667	0.00333	0.00167
BENZO(G,H,I)PERYLENE	191-24-2	2500	FDEP SCTL	830	0.00667	0.00333	0.00167
BENZO(K)FLUORANTHENE	207-08-9	1.5	FDEP SCTL	0.5	0.00667	0.00333	0.00167
CHRYSENE	218-01-9	15	FDEP SCTL	5	0.00667	0.00333	0.00167
DIBENZO(A,H)ANTHRACENE	53-70-3	0.015	FDEP SCTL	0.005	0.00667	0.00333	0.00167
FLUORANTHENE	206-44-0	1200	FDEP LEACH	400	0.00667	0.00333	0.00167
FLUORENE	86-73-7	160	FDEP LEACH	53	0.00667	0.00333	0.00167
INDENO(1,2,3-CD)PYRENE	193-39-5	0.15	FDEP SCTL	0.05	0.00667	0.00333	0.00167
PHENANTHRENE	85-01-8	250	FDEP LEACH	83	0.00667	0.00333	0.00167

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
PYRENE	129-00-0	880	FDEP LEACH	290	0.00667	0.00333	0.00167
NAPHTHALENE	91-20-3	1.2	FDEP LEACH	0.4	0.00667	0.00333	0.00167

The PAL references for soil, in hierarchical order of selection, are; FDEP SCTL: Soil Contaminant Target Level, F.A.C. 62-777 Residential Soil-Direct Table II and Leachability Based Groundwater Criteria (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept these data for decision making as long as results less than LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

Shaded and Bold row indicate the PAL is less than the DL; the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Soil
Analytical: PCBs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
AROCLOR-1016	12674-11-2	3.9	EPA RSL	1.3	0.0167	0.00833	0.00417
AROCLOR-1221	11104-28-2	0.14	EPA RSL	0.047	0.0167	0.00833	0.00417
AROCLOR-1232	11141-16-5	0.14	EPA RSL	0.047	0.0167	0.00833	0.00417
AROCLOR-1242	53469-21-9	0.22	EPA RSL	0.073	0.0167	0.00833	0.00417
AROCLOR-1248	12672-29-6	0.22	EPA RSL	0.073	0.0167	0.00833	0.00417
AROCLOR-1254	11097-69-1	0.22	EPA RSL	0.073	0.0167	0.00833	0.00417
AROCLOR-1260	11096-82-5	0.22	EPA RSL	0.073	0.0167	0.00833	0.00417

The PAL references for soil, in hierarchical order of selection, are; FDEP SCTL: Soil Contaminant Target Level, F.A.C. 62-777 Residential Soil-Direct Table II and Leachability Based Groundwater Criteria (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, 2011).

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Shaded and Bold row indicate the PAL is less than the DL; the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Soil

Analytical: TAL Metals (and *Waste Oil” Metals)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
ALUMINIUM	7429-90-5	80000	FDEP SCTL	27000	40	20	10
ANTIMONY	7440-36-0	5.4	FDEP LEACH	1.8	2.00	1.6	0.8
ARSENIC*	7440-38-2	2.1	FDEP SCTL	0.7	2	1.2	0.6
BARIUM	7440-39-3	120	FDEP SCTL	40	8	2	1
BERYLLIUM	7440-41-7	63	FDEP LEACH	21	1	0.4	0.2
CADMIUM*	7440-43-9	7.5	FDEP LEACH	2.5	1	0.4	0.2
CALCIUM	7440-70-2	NC	NC	NC	1000	400	200
CHROMIUM*	7440-47-3	38	FDEP LEACH	13	2	0.8	0.4
COBALT	7440-48-4	1700	FDEP SCTL	570	2.5	2	1
COPPER	7440-50-8	150	FDEP SCTL	50	2	1.6	0.8
IRON	7439-89-6	53000	FDEP SCTL	18000	20	12	6
LEAD*	7439-92-1	400	FDEP SCTL	130	1	0.6	0.3
MERCURY	7439-97-6	2.1	FDEP LEACH	0.7	0.033	0.033	0.013
MAGNESIUM	7439-95-4	NC	NC	NC	1000	600	200
MANGANESE	7439-96-5	3500	FDEP SCTL	1200	3	1.2	0.6
NICKEL	7440-02-0	130	FDEP LEACH	43	2	1.2	0.6
POTASSIUM	7440-09-7	NC	NC	NC	1000	600	200
SELENIUM	7782-49-2	5.2	FDEP LEACH	1.7	2	1	0.6
SILVER	7440-22-4	17	FDEP LEACH	5.7	2	0.4	0.2
SODIUM	7440-23-5	NC	NC	NC	1000	600	200
THALLIUM	7440-28-0	2.8	FDEP LEACH	0.93	1.6	0.8	0.6
VANADIUM	7440-62-2	67	FDEP SCTL	22	2.5	2	1
ZINC	7440-66-6	26000	FDEP SCTL	8700	4	2	1

* 62-770 Waste Oil Metals

The PAL references for soil, in hierarchical order of selection, are; FDEP SCTL: Soil Contaminant Target Level, F.A.C. 62-777 Residential Soil-Direct Table II and Leachability Based Groundwater Criteria (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, 2011).

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Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a “U” qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Matrix: Soil
Analytical: TRPH (by FL-PRO)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical DL (mg/kg)
TRPH	NA	340	FDEP LEACH	110	11.3	11.3	11.3

The PAL references for soil, in hierarchical order of selection, are; FDEP SCTL: Soil Contaminant Target Level, F.A.C. 62-777 Residential Soil-Direct Table II and Leachability Based Groundwater Criteria (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, 2011).

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept these data for decision making as long as results less than LOQ are "J" qualified and discussed in the uncertainties section of the Preliminary Assessment.

Shaded and Bold row indicate the PAL is less than the DL; the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, 2004).

Please note that data will be reported at the LOQ and DL, with non-detected data being the LOD followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

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

ACTIVITIES	ORGANIZATION	DATES (MM/DD/YY)		DELIVERABLE ACTUAL SUBMITTAL
		ANTICIPATED DATE(S) OF INITIATION	ANTICIPATED DATE OF COMPLETION	
Prepare Rough Draft UFP SAP and Appendices	Tetra Tech	04/01/11	06/30/11	
Submit Rough Draft UFP SAP and Appendices	Tetra Tech	--	07/22/11	07/22/11
Navy Review	Navy	07/22/11	07/29/11	
Prepare Draft UFP SAP and Appendices	Tetra Tech	08/01/11	08/05/11	
Submit Draft UFP SAP and Appendices	Tetra Tech	---	08/05/11	08/05/11
Navy Chemist Review	Navy	08/05/11	08/31/11	
Receive Comments/Comment Resolution	Tetra Tech	09/01/11	09/13/11	
Prepare Draft Final UFP SAP and Appendices	Tetra Tech	09/01/11	09/13/11	
Submit Draft Final UFP SAP & Appendices	Tetra Tech	---	09/14/11	
Regulatory Review	FDEP	09/15/11	01/5/12	
Prepare Final UFP SAP and Appendices	Tetra Tech	01/06/11	01/16/12	
Submit Final UFP SAP & Appendices	Tetra Tech	---	01/31/12	
Mobilization and Field Investigation	Tetra Tech	02/01/12	05/31/12	
Complete Field Investigation and Demobilization	Tetra Tech	---	05/31/12	
Laboratory Analysis	TBD	02/17/12	06/29/12	
Data Validation	Tetra Tech	03/18/12	07/31/12	
Database Entry	Tetra Tech	03/18/12	07/31/12	
Prepare Rough Draft SAR	Tetra Tech	06/01/12	08/31/12	
Submit Draft SAR	Tetra Tech	---	08/31/12	
Navy Review	Navy	09/03/12	10/31/12	
Prepare Draft SAR	Tetra Tech	11/01/12	11/30/12	
Submit Draft Final SAR	Tetra Tech	---	11/30/12	
Regulator Review	MDEQ	12/03/12	01/04/13	
Receive Comments/Comment Resolution	Tetra Tech	01/07/13	02/07/13	
Prepare Final SAR	Tetra Tech	01/07/13	02/07/13	
Submit Final SAR	Tetra Tech	---	02/07/13	

Bold activities are deliverables.

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

The sampling activities to be conducted in support of the SA for Sites 103, 104, and 105 are presented below including the proposed sample locations, sampling methods, and a rationale for the sampling activities. The proposed sample locations are presented on Figures 17-1a and 17-1b. The analytical program recommended for each proposed sample is presented in Worksheet #18. Environmental data have not previously been collected at Sites 103, 104, and 105; therefore, little information is available to help determine where site-related contamination could exist. The proposed soil and groundwater sampling locations for Sites 103, 104, and 105 were chosen based on the Project Team's understanding of the location of former site features and buildings, the CSM, the current understanding of site-specific conditions, and the need to collect data that will help resolve the problem described in Worksheet #11.

Surface and Subsurface Soil Sampling

During the initial round of sampling, surface and subsurface soil data will be collected from 44 soil borings at Site 103, from 48 soil borings at Site 104, and from 20 soil borings at Site 105. Soil samples will be collected using hand auger from the land surface to a minimum of 4 feet bgs in order to clear utilities, then by DPT to 10 feet bgs or just above the water table, whichever is encountered first. Up to two intervals (one surface and one subsurface) will be collected for fixed-base laboratory analysis based on field screening with a FID/PID (maximum detected screening values) or visual/olfactory inspection (oily residues and smells). Surface soil will consist of soil from the land surface to a depth of 2 feet, and subsurface soil will consist of soil from 2 feet bgs to the water table.

Surface soil samples will be collected in two intervals- 0-6 inches bgs and 6-24 inches bgs. The 0-6 inch interval sample will be analyzed for all target analyte groups but VOCs. All target analytes will be measured in the 6-24 inch interval sample. In accordance with Chapter 62-780, F.A.C., subsurface soil samples shall be collected at 2-foot intervals beginning at 2 feet bgs unless the sampling intervals are adjusted, as necessary, to account for factors such as discrete variations in the lithology, depth to the water table, the point of discharge, and the chemical and physical properties of the contaminants. Sampling intervals may also be adjusted as a result of a known or suspected surficial discharge of metals or SVOCs (based on site history and visual/olfactory inspection [oily residues and smells] in the field).

If no contamination is detectable using field screening or visual and/or olfactory observations at a particular location, soil must be collected from the shallowest 2 feet (i.e., surface soil) and the 2-foot interval immediately above the water table smear zone.

Soil boring locations are identified on Figures 17-1a and 17-1b, but may be relocated by the Tetra Tech FOL, with the concurrence of the Tetra Tech PM based on field observations, physical obstructions, or utilities.

Water Level Measurements

A synoptic round of electronic water-level measurements will be conducted at Sites 103, 104, and 105 as part of each groundwater sampling event to provide information regarding groundwater flow patterns and hydraulic gradients. Synoptic water-level measurements will be completed prior to sampling and within the shortest time possible on the same day and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data sets.

Groundwater Sampling

During the surface and subsurface soil sampling, groundwater will be collected from each of the DPT borings for submittal to an onsite mobile laboratory for VOC analysis. The DPT groundwater samples will be withdrawn from the temporary well point via polyethylene tubing to the surface via a peristaltic pump and placed in the sample container via the straw method. DPT well locations are identified on Figures 17-1a and 17-1b, but proposed well locations may be relocated by the Tetra Tech FOL with the concurrence of the Tetra Tech PM based on field observations, physical obstructions, or utilities.

Based on the results of the DPT soil and groundwater investigation, multiple shallow and deep monitoring wells will be installed at Site 103, 104, and 105 as described below:

- Site 103 – 22 shallow and 3 deep monitoring wells
- Site 104 – 24 shallow and 3 deep monitoring wells
- Site 105 – 10 shallow and 3 deep monitoring wells

Groundwater samples will be collected from permanent monitoring wells utilizing a peristaltic pump. Purging and sampling will be conducted using the FDEP low-flow purging techniques (discharge rate of less than 1 liter per minute).

Site Specific Background

The field screening data will be used to select a site specific background location for each Site. The sample location that is selected by the Project Team to be the site specific background will be located hydraulically upgradient of each Site and based on the field screening data will not contain site related contaminants. Background soil and groundwater samples will be collected from each site specific background location. Background soil samples will be collected from land surface to 6 inches (excluding VOCs), 6 inches to 2 feet, and, thereafter, at 2-foot intervals. The background groundwater sample will

be from a shallow monitoring well that is screened across the surficial water table at is at an estimated depth of approximately 15 feet bgs.

UST Determination

The presence of the five USTs at Site 103 will be confirmed during the field investigation using magnetic and ferrous metal detectors. Because the presence of the USTs at Site 103 is not definitively known, a Schonstedt MAC 51 BX Pipe and Cable Locator will be used to locate the USTs using the existing pipe line, assuming that they are still connected to the USTs. This instrument creates a magnetic field that is used to locate conductive features such as the USTs and pipeline by connecting a transmitter to one end of a metal pipe. A hand held detector picks up the enhanced magnetism of the pipeline and USTs allowing them to be easily located. An additional ferrous metal detector instrument, the Schonstedt XTpc will be used to aid in the location of the USTs. Hand auger borings and probing with a metal rod will also be conducted to confirm if the USTs are still in place.

The presence of USTs at Sites 104 and 105 will be confirmed during the field investigation using a ferrous metal detector. Because there is not known piping at the land surface for the USTs at Sites 104 and 105, a ferrous metal detector instrument, the Schonstedt XTpc will be used to aid in the location of the USTs. Hand auger borings and probing with a metal rod will also be conducted to confirm if the USTs are still in place.

General Sampling and Analysis

Proposed sampling locations may be revised by the Tetra Tech FOL with the concurrence of the Tetra Tech PM based on field screening, site observations, or site conditions. Field QC samples will be collected as part of the investigation, including field duplicates, trip blanks, and equipment rinsate blanks. Worksheet # 20 presents the field QC sample summary. Also, additional sample volume will be collected as necessary for the laboratory QC of matrix spike/matrix spike duplicate (MS/MSD) analyses (VOCs, SVOCs [including low level PAHs], PCBs, and TRPH) and MS/laboratory duplicate analyses (for metals). The target analytes associated with the surface soil, subsurface soil, and groundwater samples are presented in Worksheet #15. The Analytical Method/SOPs are identified in Worksheet #23.

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

Sampling Location/Identification Number ⁽¹⁾	Matrix	Depth/Location (feet bgs)	Analytical Group	Number of Samples	Sampling SOP Reference
SOIL SAMPLES – SITES 103, 104, and 105					
BF-103-SB01- XX-YYYYMMDD through BF-103-SB44-XX-YYYYMMDD	Soil	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), Waste Oil Metals, and TRPH	88	FDEP FS 3000
BF-104-SB01- XX-YYYYMMDD through BF-104-SB48-XX-YYYYMMDD	Soil	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	96	FDEP FS 3000
BF-105-SB01- XX-YYYYMMDD through BF-105-SB20-XX-YYYYMMDD	Soil	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	40	FDEP FS 3000
DPT GROUNDWATER SAMPLES – SITES 103, 104, and 105					
BF-103-GW01- XX-YYYYMMDD through BF-103-GW44-XX-YYYYMMDD	Groundwater	TBD	VOCs	44	FDEP FS 2200
BF-104-GW01- XX-YYYYMMDD through BF-104-GW48-XX-YYYYMMDD	Groundwater	TBD	VOCs	48	FDEP FS 2200
BF-105-GW01- XX-YYYYMMDD through BF-105-GW20-XX-YYYYMMDD	Groundwater	TBD	VOCs	20	FDEP FS 2200
PERMANENT MONITORING WELL GROUNDWATER SAMPLES – SITES 103, 104, and 105					
BF-103-MW01- XX-YYYYMMDD through BF-103-MW25-XX-YYYYMMDD	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), Waste Oil Metals, and TRPH	25	FDEP FS 2200
BF-104-MW01- XX-YYYYMMDD through BF-104-MW27-XX-YYYYMMDD	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	27	FDEP FS 2200
BF-105-MW01- XX-YYYYMMDD through BF-105-MW13-XX-YYYYMMDD	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	13	FDEP FS 2200
FIELD DUPLICATES – SOIL					
BF-103-SB-YYYYMMDD-FD01 through BF-103-SB-YYYYMMDD-FD09	Soil	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), Waste Oil Metals, and	9	FDEP FS 3000

Sampling Location/Identification Number ⁽¹⁾	Matrix	Depth/ Location (feet bgs)	Analytical Group	Number of Samples	Sampling SOP Reference
			TRPH		
BF-104-SB-YYYYMMDD-FD01 through BF-104-SB-YYYYMMDD-FD09	Soil	TBD	VOCs (plus TICs, SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	9	FDEP FS 3000
BF-105-SB-YYYYMMDD-FD01 through BF-105-SB-YYYYMMDD-FD04	Soil	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	4	FDEP FS 3000
FIELD DUPLICATES – DPT GROUNDWATER					
BF-103-GW-YYYYMMDD-FD01 through BF-103-GW-YYYYMMDD-FD09	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), Waste Oil Metals, and TRPH	9	FDEP FS 2200
BF-104-GW-YYYYMMDD-FD01 through BF-104-GW-YYYYMMDD-FD09	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	9	FDEP FS 2200
BF-105-GW-YYYYMMDD-FD01 through BF-105-GW-YYYYMMDD-FD04	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	4	FDEP FS 2200
FIELD DUPLICATES – GROUNDWATER PERMENANT MONITORING WELLS					
BF-103-GW-YYYYMMDD-FD01 through BF-103-GW-YYYYMMDD-FD03	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), Waste Oil Metals, and TRPH	3	FDEP FS 2200
BF-104-GW-YYYYMMDD-FD01 through BF-104-GW-YYYYMMDD-FD03	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	3	FDEP FS 2200
BF-105-GW-YYYYMMDD-FD01	Groundwater	TBD	VOCs (plus TICs), SVOCs (including low level PAHs and TICs), PCBs, TAL Metals, and TRPH	1	FDEP FS 2200

- XX – Sample Depth – Bottom of sample interval in feet below ground surface (bgs)
 ZZ – Indicates Well identifier will be determined in the field for confirmatory groundwater samples.
 YYYYMMDD – date of sample – year, month, date

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

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference ¹	Sample Size	Containers (number, size, and type) ²	Preservation Requirements	Maximum Holding Time ⁽³⁾ (preparation / analysis)
Soil	VOCs (plus TICs)	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram Encore samplers or terracores	5 grams	Sodium bisulfate $\leq 6^{\circ}\text{C}$; or in water and freeze to $< -10^{\circ}\text{C}$	48 hours from sampling to preparation, 14 days to analysis
Groundwater and aqueous QC samples	VOCs (plus TICs)	SW-846 5030/8260B Empirical SOP-202	Three 40- mL glass vials	5 mL	Hydrochloric acid (HCl) to pH<2; Cool to $\leq 6^{\circ}\text{C}$; no headspace	14 days to analysis
Groundwater and aqueous QC samples	1,2-Dibromoethane	SW-846 5030/8011 Empirical SOP-218	Three 40-mL glass vials	5 mL	HCl to pH<2; Cool to $\leq 6^{\circ}\text{C}$; no headspace	14 days to analysis
Groundwater	VOCs Screening Level Data	SW-846 8260B SOP09001R0 and 11-001R0	Two 40 mL glass vials	5 mL	HCl to pH<2; Cool to 0 to 6°C ; no headspace	14 days to analysis
Soil	SVOCs (including low level PAHs and TICs)	SW-846 3546/8270C Empirical SOP-201/343	One 4-ounce (oz) glass jar	15 grams	Cool to $< 6^{\circ}\text{C}$	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	SVOCs (including low level PAHs and TICs)	SW-846 3510C/3520/8270C Empirical SOP-201/300	Two 1 - liter (L) glass amber bottles	1,000 mL	Cool to $< 6^{\circ}\text{C}$	7 days until extraction, 40 days to analysis
Soil	PCBs	SW-846 3540/3545/3550/8082A, Empirical SOP-211/343	One 4-ounce glass jar	30 grams	Cool to $\leq 6^{\circ}\text{C}$	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	PCBs	SW-846 3510C/3520/8082A, Empirical SOP-211/302	Two 1-Liter glass amber bottles	1,000 mL	Cool to $\leq 6^{\circ}\text{C}$	7 days until extraction, 40 days to analysis
Soil	TAL Metals and Waste Oil Metals	SW-846 3050B/6010C Empirical SOP-100/104/105	One 4-ounce glass jar	1 to 2 grams	Cool to $\leq 6^{\circ}\text{C}$	180 days to analysis except mercury, 28 days for mercury
Groundwater and aqueous QC samples	TAL Metals and Waste Oil Metals	SW-846 3010A/6010C Empirical SOP-100/103/105	One 500-mL plastic bottle	50 mL	Nitric acid to pH <2; Cool to $\leq 6^{\circ}\text{C}$	180 days to analysis except mercury, 28 days for mercury
Soil	TRPH	FL-PRO Empirical SOP-338/343	One 4-ounce glass jar	15 grams	Cool to $\leq 6^{\circ}\text{C}$	14 days until extraction, 40 days to analysis

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference ¹	Sample Size	Containers (number, size, and type) ²	Preservation Requirements	Maximum Holding Time ⁽³⁾ (preparation / analysis)
Groundwater and aqueous QC samples	TRPH	FL-PRO Empirical SOP-338	Two - 1L amber glass	1,000 mL	HCl to pH <2; Cool to < 6 °C	7 days until extraction, 40 days to analysis
IDW ⁴	TCLP Organics	SW-846 1311/3510C/5030B/8260 B/8270D/8081B/8151A/	4, 8oz jars	400 grams	Cool to < 6 °C	14 days leach/14 days analysis
IDW ⁴	TCLP Inorganics	SW-846 1311/3010/6010C/ 7470A	1, 8oz jar	100 grams	Cool to < 6 °C	180 days leach/28 days mercury leach & analysis

Notes:

mL = milliliter

TCLP = Toxicity Characteristic Leaching Procedure

- 1 Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.
- 2 Sample size is a minimum; the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring MS/ MSD, containers listed should be tripled.
- 3 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
- 4 Soil IDW sample analyses are presented on this worksheet for the utilization of field personnel. QC information is not presented in any of the remaining worksheets as these samples are for waste disposal, not decision making purposes. IDW sample analytical results will not be validated.

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

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs		Number of Equip. Blanks	Number of VOA Trip Blanks	Total Number of Samples to Lab
Soil Samples	VOCs (plus TICs)	224	23	12		12	10	269
	1,2-Dibromoethane	224	23	12		12	0	259
	SVOCs (including low level PAHs and TICs)	224	23	12		12	NA	259
	PCBs	136	14	7		7	NA	157
	TAL Metals	136	14	7		7	NA	157
	Waste Oil Metals	88	9	4		4	NA	101
	TRPH	224	23	12		12	NA	259
Mobile Lab Groundwater Samples	VOCs	112	11	5		5	5	133
Permanent Monitoring Well Groundwater Samples	VOCs (plus TICs)	65	7	3		3	3	78
	1,2-Dibromoethane	65	7	4		4	0	76
	SVOCs (including low level PAHs and TICs)	65	7	3		3	NA	75
	PCBs	65	7	3		3	NA	75
	TAL Metals	40	4	2		2	NA	46
	Waste Oil Metals	25	3	1		1	NA	29
	TRPH	65	7	3		3	NA	75

Notes:

VOA = Volatile organic analysis

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

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

SAP Worksheet #21 – Project Sampling SOP References Table

REFERENCE NUMBER	TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
FC 1000	Title: Cleaning/Decontamination Procedures Revision: <u>December 3, 2008</u>	FDEP	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate-free detergent, deionized water	N	Contained in Appendix B
FD 1000	Title: Field Documentation Procedures Revision: <u>December 3, 2008</u>	FDEP	Log book	N	Contained in Appendix B
FM 1000	Title: Field Mobilization Procedures Revision: <u>December 3, 2008</u>	FDEP	Not Applicable (NA)	N	Contained in Appendix B
FQ 1000	Title: Field Quality Control Procedures Revision: <u>December 3, 2008</u>	FDEP	NA	N	Contained in Appendix B
FS 2200	Title: Groundwater Sampling Procedure Procedures Revision: <u>December 3, 2008</u>	FDEP	Peristaltic Pump, Tubing, Flow through cells, sample log sheets	N	Contained in Appendix B
FS 3000	Title: Soil Sampling Procedure Procedures Revision: <u>December 3, 2008</u>	FDEP	Sample log sheets, boring logs	N	Contained in Appendix B
FT 1000	Title: Field Testing General Revision: <u>December 3, 2008</u>	FDEP	NA	N	Contained in Appendix B
FT 1100	Title: Field pH Revision: <u>December 3, 2008</u>	FDEP	pH Meter, calibration log	N	Contained in Appendix B
FT 1200	Title: Field Specific Conductance Revision: <u>December 3, 2008</u>	FDEP	Conductivity Meter, calibration log	N	Contained in Appendix B
FT 1400	Title: Field Temperature Revision: <u>December 3, 2008</u>	FDEP	Thermometer, calibration log	N	Contained in Appendix B
FT 1500	Title: Field Dissolved Oxygen Revision: <u>December 3, 2008</u>	FDEP	Dissolved Oxygen Probe, calibration log	N	Contained in Appendix B
FT 1600	Title: Field Turbidity Revision: <u>December 3, 2008</u>	FDEP	Turbidity Meter, calibration log	N	Contained in Appendix B

REFERENCE NUMBER	TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
SA-2.5	Title: <u>Direct Push Technology (Geoprobe®/Hydropunch™)</u> Effective Day: <u>September, 2003</u> <u>Revision 3</u>	Tetra Tech	Geoprobe and sampling equipment	N	Contained in Appendix B
SA-7.1	Title: <u>Management of Investigation Derived Waste</u> Revision: <u>November 1, 2007</u> Number: <u>SESDPROC-202-R1</u>	Tetra Tech	NA	N	Contained in Appendix B
CT-04	Title: <u>Sample Nomenclature</u> Effective Day: <u>March 9, 2009</u> <u>Revision 2</u>	Tetra Tech	NA	N	Contained in Appendix B
CT-05	Title: <u>Database Record and Quality Assurance</u> Effective Day: <u>January 29, 2001</u> <u>Revision 2</u>	Tetra Tech	NA	N	Contained in Appendix B
GH-1.2	Title: <u>Evaluation of Existing Monitoring Wells and Water Level Measurement</u> Effective Day: <u>September 2003</u> <u>Revision 2</u>	Tetra Tech	NA	N	Contained in Appendix B
GH-1.5	Title: <u>Borehole and Sample Logging</u> Effective Day: <u>June 1999</u> <u>Revision 1</u>	Tetra Tech	NA	N	Contained in Appendix B
GH-2.8	Title: <u>Groundwater Monitoring Well Installation</u> Effective Day: <u>September 2003</u> <u>Revision 3</u>	Tetra Tech	Health and safety equipment, well drilling and installation equipment, hydrogeologic equipment, drive point installation tools	N	Contained in Appendix B
HS-1.0	Title: <u>Utility Locating</u> Effective Day: <u>September 2003</u> <u>Revision 3</u>	Tetra Tech		N	Contained in Appendix B

REFERENCE NUMBER	TITLE, REVISION DATE AND/OR NUMBER	ORIGINATING ORGANIZATION OF SAMPLING SOP	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
DV-01	Title: <u>Data Validation- Contract Laboratory Program (CLP) Organics for Solid and Aqueous Matrices</u> Effective Day: <u>January 28, 2009</u> <u>Revision 3</u>	Tetra Tech	NA	N	Contained in Appendix B
DV-03	Title: <u>Data Validation- CLP Inorganics for Solid and Aqueous Matrices</u> Effective Day: <u>February 2, 2009</u> <u>Revision 0</u>	Tetra Tech	NA	N	Contained in Appendix B

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

Notes:

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

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

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

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM? (Y/N)	Modified for Project Work? ¹ (Y/N)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 21, 09/01/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals digestion	NA/ Preparation	Empirical	N	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique Methods SW846 7470A and 245.1, CLP-M 4.1 (Revision 18, 04/11/10)	Definitive	Groundwater and aqueous QC samples/ Mercury	Flow Injection Mercury Analyzer	Empirical	N	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique Methods SW846 7471A, 7471B, 245.5, and CLP-ILM 4.1 (Revision 19, 04/11/10)	Definitive	Soil/ Mercury	Flow Injection Mercury Analyzer	Empirical	N	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19 th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals	ICP -AES	Empirical	N	N
Empirical SOP-201	Gas Chromatography Mass Spectrometry (GC/MS) Semivolatiles and Low-Concentration PAHs by Method 625 and SW846 Method 8270C and 8270D, including Appendix IX Compounds (Revision 20, 04/26/10)	Definitive	Soil, groundwater, and aqueous QC samples/ SVOCs (including low level PAHs)	GC/MS	Empirical	N	N
Empirical SOP-202	GC/MS Volatiles by USEPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 23, 09/09/10)	Definitive	Soil, groundwater, and aqueous QC samples/ VOCs	GC/MS	Empirical	N	N

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM? (Y/N)	Modified for Project Work? ¹ (Y/N)
Empirical SOP-211	Gas Chromatography/ Electron Capture Detector (GC/ECD) Organochlorine Pesticides/ PCBs using USEPA Method 608/608.2 or SW846 Method 8081A/8082 or 8081B/8082A (Revision 22, 07/07/10)	Definitive	Soil, groundwater, and aqueous QC samples/PCBs	GC/ECD	Empirical	N	Y concentrate final extract volume to 2mL
Empirical SOP-218	1,2-Dibromoethane and 1,2-dibromo-3-chloropropane by GC/ECD using USEPA Method 504.1 and SW-846 8011, (Revision 7, 09/07/10).	Definitive	Groundwater and aqueous QC samples/1,2-Dibromoethane	GC/ECD	Empirical	N	N
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction using SW-846 Method 5035 for 8260B Analysis (Revision 9, 9/07/10)	Definitive	Soil / VOCs extraction	NA/ Extraction	Empirical	NA	N
Empirical SOP-300	GC/MS Semivolatile Base/Neutral/Acid (BNA)-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis (Revision 18, 04/23/10)	Definitive	Groundwater and aqueous QC samples/ SVOCs extraction	NA/ Extraction	Empirical	NA	N
Empirical SOP-302	Pesticide/PCBs, Aqueous Matrix Extraction for EPA 608/608.2 and SW846 Method 8081A/8082 Using Method 3510C (Revision 17, 04/26/10)	Definitive	Groundwater and aqueous QC samples/ PCBs extraction	NA/ Extraction	Empirical	NA	N
Empirical SOP-343	BNA, Pesticide/PCB, and TPH Non-aqueous Matrix (Microwave Extraction) using SW-846 Method 3546 (Revision 1, 09/09/10)	Definitive	Soil / SVOCs, PCBs Extraction	NA/ Extraction	Empirical	NA	N
Empirical SOP-338	FL-PRO for Determination of Petroleum Range Organics (Revision 8, 04/29/10)	Definitive	Soil, Groundwater, and Aqueous Field QC Samples/ TRPH	Gas Chromatography/Flame Ionization Detector (GC/FID)	Empirical	NA	N
09001R0	Analysis of Selected VOCs by GS/MS in Water and Soil Matrices, Revision 0, 4/21/09.	Screening	Groundwater - VOCs	GC/MS	ALF	NA	N

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

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
GC/MS VOCs plus TICs	Initial Calibration (ICAL) - A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters); or if the daily calibration fails.	The average Response Factors (RFs) for System Performance Check Compounds (SPCCs): 1,1,2,2-tetrachloroethane and chlorobenzene must be ≥ 0.30 ; chloromethane, 1,1-Dichloroethane and bromoform must be ≥ 0.10 ; The Percent Relative Standard Deviations (%RSDs) for RFs of Calibration Check Compound (CCCs) must be $\leq 30\%$; and the Relative Standard Deviations (RSDs) must be $\leq 15\%$ for all compounds. If not met: Option 1) Linear least squares regression: Linear Regression Correlation Coefficient (r) must be ≥ 0.995 ; or Option 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.990 (6 points are required for second order).	Repeat calibration if criterion is not met. Samples may be analyzed using an ICAL in which one or two target analytes do not meet %RSD or regression criteria provided that adequate sensitivity is evident at the LOQ. If the affected analyte(s) are not detected in the associated field samples, no corrective action is necessary. If any affected analyte is detected in an associated field sample, the sample must be reanalyzed under a passing ICAL.	Analyst, Department Manager	Empirical SOP-202
	Retention Time (RT) Window Position Establishment	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 Continuing Calibration Verification (CCV) is used.	NA.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
GC/MS VOCs plus TICs	Initial Calibration Verification (ICV) – approximately mid-range standard of a source different than that used to prepare the ICAL standards (Second Source)	Once after each ICAL, prior to sample analysis.	Percent Recovery (%R) must be within 80-120% of true value for all project compounds.	Correct problem and verify ICV. Reanalyze ICV and/or ICAL as appropriate. If a compound fails the acceptance criteria with a higher than expected response up to 40 percent difference (%D) (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 2 compounds).	Analyst, Department Manager	Empirical SOP-202
	CCV	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune and before sample analysis.	%D must be $\leq 20\%$ for all project compounds and surrogates. The RFs for SPCCs must be ≥ 0.10 & ≥ 0.30 (compounds as listed above in ICAL block).	Investigate cause and repeat injection once. If failure repeats, repeat ICAL and reanalyze all samples analyzed since the last successful CCV. If a compound fails the acceptance criteria with a higher than expected response up to 40%D (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 2 compounds).	Analyst, Department Manager	
	BFB Tune	Prior to ICAL and at the beginning of each 12 hour analytical sequence.	Must meet the ion abundance criteria required by the method.	Retune and/or clean source. No samples may be analyzed without a valid tune.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
GC/MS VOCs (and Selected Ion Monitoring 1,4-dioxane) screening by mobile lab	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Prior to any sample analysis.	The %RSD of target analyte RFs must be $\leq 20\%$. Minimum mean RFs of SPCCs as listed in SW-846 8260B must be met during the ICAL. The %RSDs of CCC RFs during ICAL must be $< 30\%$.	Correct problem and repeat ICAL. Do not analyze samples until ICAL passes criteria.	Analyst	SOP 09001R0 and 11-001R0
	CCV – A midlevel standard run every 12 hours prepared from separate source from calibration standards	Daily before sample analysis and every 12 hours of analysis time.	RF criteria for SPCCs the same as during ICAL. RF of CCCs must be $\leq 20\%$ D from ICAL.	Rerun CCV. Then rerun ICAL, if necessary.	Analyst	
	BFB Tune	Prior to ICAL and at the beginning of each 12-hour analytical sequence.	Criteria listed in Section 18.3 of SOP 09001R0 and 11-001R0.	Retune and/or clean source.	Analyst	
GC/ECD 1,2-Dibromoethane	ICAL - Minimum five-point calibration is required	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	One of the options below: Option 1: RSD must be $\leq 20\%$ Option 2: linear least squares regression: r must be ≥ 0.995 Option 3: non-linear regression: coefficient of determination r^2 must be ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Analyst, Department Manager	Empirical SOP -218
	ICV	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within $\pm 20\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	%D must be within $\pm 20\%$ D 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.	Analyst	
GC/MS SVOCs (including low level PAHs and TICs)	Breakdown Check (dichlorodiphenyl trichloroethane [DDT] only)	At the beginning of each 12-hour analytical sequence.	The degradation must be $\leq 20\%$ for DDT to verify inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is $\leq 20\%$ for DDT.	Analyst, Department Manager	Empirical SOP-201
	ICAL – A minimum 5-point calibration is required	Calibrate the instrument when it is received, after a major change (source cleaning, new column, change in GC run parameters); or if the daily calibration fails.	Average RF SPCCs must be ≥ 0.050 ; %RSD for RFs for CCCs must be $\leq 30\%$; and the %RSD must be $\leq 15\%$ for all other compounds. If not met: Option 1) r must be ≥ 0.995 , or Option 2) r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Samples may be analyzed using an ICAL in which one or two target analytes do not meet %RSD or regression criteria provided that adequate sensitivity is evident at the LOQ. If the affected analyte(s) are not detected in the associated field samples, no corrective action is necessary. If any affected analyte is detected in an associated field sample, the sample must be reanalyzed under a passing ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	ICV – Second Source	Once after each ICAL prior to sample analysis	%D must be ≤ 20% for all project compounds. SPCC RFs must be ≥ 0.050.	Correct problem and verify second source standard. Reanalyze ICV and/or ICAL as appropriate. If a compound fails the acceptance criteria with a higher than expected response up to 40%D (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 2 compounds).	Analyst, Department Manager	
GC/MS SVOCs (including low level PAHs and TICs)	RT Window Position Establishment	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.	NA.	Analyst, Department Manager	Empirical SOP-201
	Evaluation of RRTs	With each sample.	RRT of each target analyte must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl-phosphine (DFTPP) tune and before sample analysis.	%D must be $\leq 20\%$ for all project compounds and surrogates. SPCCs RFs must be >0.050 .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data. If a compound fails the acceptance criteria with a higher than expected response up to 40%D (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 2 compounds)	Analyst, Department Manager	
	DFTPP Tune	Prior to ICAL and at the beginning of each 12 hour analytical sequence.	Must meet the ion abundance criteria required by the method.	Retune and/or clean source. No samples may be analyzed without a valid tune.	Analyst, Department Manager	
GC/ECD PCBs	ICAL - A minimum 5-point calibration curve is run for Aroclor 1016 and 1260 and a single-point reference for all other Aroclors. If an Aroclor other than 1016/1260 is identified in any sample by peak pattern, then the sample is re-analyzed with a full calibration curve for that Aroclor	Instrument receipt, major instrument change, when CCV does not meet criteria.	%RSD for each analyte must be $\leq 20\%$ If not met: Option 1) r must be ≥ 0.995 , or Option 2) r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	Empirical SOP-211

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	ICV – Second Source	Once after each ICAL and prior to sample analysis	%R of all project compounds must be within 80-120% of true value.	Identify source of problem, correct, repeat calibration, rerun samples. If a compound fails the acceptance criteria with a higher than expected response up to 30%D (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 1 compound).	Analyst, Department Manager	
GC/ECD PCBs	CCV	Once after each ICAL and at the beginning and end of each run sequence and every 10 samples.	%D of all project compounds must be ≤ 20%.	Identify source of problem, correct, repeat calibration, rerun samples. If a compound fails the acceptance criteria with a higher than expected response up to 30%D (indicating a high bias), and that compound is not detected above the LOQ in any associated field sample, no corrective action is necessary (limited to 1 compound).	Analyst, Department Manager	Empirical SOP-211
ICP-AES TAL and Waste Oil Metals	ICAL - the instrument is calibrated by a 1-point calibration per manufacturer's guidelines	At the beginning of each day, or if the QC is out of criteria.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP-100/105

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R of all project compounds must be within 90-110% of the true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration.	Analyst, Department Manager	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No project compounds detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	The %R of all project compounds must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No project compounds detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst, Department Manager	
	Low-Level Check Standard (if using 1-point ICAL)	Daily after 1-point ICAL and before samples.	The %R of all project compounds must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
	Interference Check Standards (ICS) – ICS A and ICS B)	At the beginning and end of an analytical run and after each batch of 20 samples.	The absolute value of ICS A recoveries for non-spiked analytes must be \leq LOD; and ICS B %Rs must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
Cold Vapor Mercury Analyzer	ICAL – a 6-point calibration curve is analyzed	Daily prior to sample analysis, and if continuing QC fails.	The RSD for RFs must be \leq 20%, or r must be \geq 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	Empirical SOP-103/104
	ICB and CCB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, re-prepare, and reanalyze.	Analyst, Department Manager	
	ICV - Second Source	Once after each ICAL and prior to sample analysis	%R for mercury must be within 90-110%.	Correct problem and repeat calibration.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	CCV	CCV-at beginning and end of each run sequence and every 10 samples.	%R for mercury must be within 80-120%.	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Department Manager	
GC/FID TRPH	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Perform after major instrument maintenance and upon failure of second consecutive CCV, prior to sample analysis.	The average %RSD for all 17 RFs must be $\leq 20\%$, If not met: Option 1) r must be ≥ 0.995 . Option 2) 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	Empirical SOP-338
	ICV – Second Source	After each ICAL, prior to the analysis of samples.	The %R must be within 75-125% of the true value.	Determine problem and recalibrate.	Analyst, Department Manager	
	CCV	At the beginning of a sequence and after every 12 hours or 10 samples. (whichever comes first), then at the end of the sequence.	The %R must be within 75-125% of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

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

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Check pressure, gas supply and vacuum 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, clean MS source as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	VOCs plus TICs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-202
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	1,2-dibromoethane	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Supervisor	Empirical SOP-218
GC/MS	Check pressure, gas supply, and vacuum daily. Bake out column, manual tune if DFTPP not in criteria, change septa as needed, cut column as needed, clean MS source as needed. Other maintenance specified in lab Equipment Maintenance SOP.	SVOCs (including low level PAHs and TICs)	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-201
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-211
ICP-AES	Clean sample path, check pump tubing, argon level, vacuum and waste container daily. Clean source as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	TAL and Waste Oil Metals (except mercury)	Pump, pump tubing, vacuum source, and waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-105

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell, waste container.	Prior to ICAL and as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-103
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in laboratory SOPs	TRPH (FL-PRO)	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-338

Notes: ¹ Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

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

Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): FOL or designee/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/ Tetra Tech
Type of Shipment/Carrier: Federal Express
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Sample Custodians/ Empirical and ALF
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical and ALF
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory/ Empirical
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory/ Empirical and ALF
Sample Archiving
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/ Digestate Storage (Number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): NA
Sample Disposal
Personnel/Organization: Sample Custodians/ Empirical and ALF

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

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

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

Sample Identification

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

Sample Collection Documentation

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

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

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

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

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

Sample Handling and Tracking System

Following sample collection using the appropriate bottleware, samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottleware during shipment. The cooler will be secured using strapping/packaging tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at ≤ 6 °C until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

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

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

Sample Handling

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

Sample Delivery

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

Sample Custody

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

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

Laboratory Custody

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical SOPs. Coolers are received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody form will be checked against the sample containers for accuracy. Samples will be logged into the Laboratory Information Management System and given a unique log number which can be tracked through processing. The Laboratory PM will notify the Tetra Tech FOL verbally or via e-mail of any problems on the same day that an issue is identified.

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

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	VOCs plus TICs					
Analytical Method/SOP Reference	SW-846 8260B/ Empirical SOP-202					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples.	All target compounds must be $\leq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	Correct problem, If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Department Manager	Contamination/ Bias	Same as QC Acceptance Limits.
Laboratory Control Sample (LCS) Laboratory Control Sample Duplicate (LCSD) (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. If LCSD performed - 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 analytes, if sufficient sample material is available. Refer to DoD QSM Version 4.1 Table G-1 for number of marginal exceedances allowed. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Department Manager	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	VOCs Plus TICs					
Analytical Method/SOP Reference	SW-846 8260B/ Empirical SOP-202					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS/MSD	One per SDG or every 20 samples of similar matrix.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager	Accuracy / Bias Precision	Same as QC Acceptance Limits.
Surrogate	Every field and QC sample. Four per sample: Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Department Manager	Accuracy / Bias	Same as QC Acceptance Limits.
Internal Standard (IS)	Every field sample, standard, and QC sample. Three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs for ISs must be within ± 30 seconds and the response areas must be within -50% to +100% of the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Department Manager	Accuracy/ Bias	Same as QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results detected between DL and LOQ.	NA.	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per daily analysis batch.	No analytes > 1/2 of the LOQ.	Bake out purge and trap system, change adsorbent trap. Re-prepare and reanalyze method blank and associated samples.	Analyst	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: dibromofluoromethane 1,2-dichloroethane-d ₄ Toluene-d ₈ BFB	Should be within limits established by lab or method.	Reanalyze sample. If one or more still remain outside criteria, then recalibrate and/or remake surrogate solution.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix.	Should be within limits established by lab.	Check LCS to see if matrix effects apply.	Analyst	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.
LCS	One per daily analysis batch.	%R must be between 70 to 130.	Re-prepare and reanalyze LCS. Reanalyze associated samples.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Three per sample- Fluorobenzene Chlorobenzene-d ₅ 1,4-dichlorobezene-d ₄	RTs for ISs must be within ± 0.1 minute and the response areas must be within -50% to +100% of the last calibration check.	Reanalyze sample. If one or more still remain outside criteria, recalibrate.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Groundwater and Aqueous QC samples					
Analytical Group	1,2-Dibromoethane					
Analytical Method/SOP Reference	SW-846 8011/ Empirical SOP-218					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix.	All target analytes must be $\leq \frac{1}{2}$ LOQ.	Correct problem, If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Department Manager	Bias/ Contamination	Same as QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs must be between 70% - 130%. If LCSD performed - The RPD must be \leq 30%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Department Manager	Accuracy/ Bias	Same as QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix (spike same as LCS).	%Rs must be between 70% - 130%. The RPD must be \leq 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be $RPD \leq 40\%$. Report the higher of the two concentrations, unless there is interference.	None. Apply "P" flag if $RPD > 40\%$ and discuss in the case narrative.	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.
Results between DL and LOQ	NA.	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	SVOCs (including low level PAHs and TICs)					
Analytical Method / SOP Reference	SW-846 8270C/ Empirical SOP-201					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	All target compounds must be $\leq \frac{1}{2}$ LOQ except common laboratory contaminants, which must be $<$ LOQ.	(1) Investigate source of contamination (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank.	Analyst, Department Manager	Bias/ Contamination	Same as QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD \leq 30% (for LCS/LCSD, if LCSD is analyzed)	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to DoD QSM Version 4.1 Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Department Manager	Accuracy/Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples.	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD \leq 30%.	Corrective action will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	SVOCs (including low level PAHs and TICs)					
Analytical Method / SOP Reference	SW-846 8270C/ Empirical SOP-201					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	Every field and QC sample. Six per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, then check instrument performance; if problem is found, then correct and reanalyze. (3) If still out, then re-extract and analyze sample. (4) If reanalysis is out, then flag data.	Analyst, Department Manager	Accuracy/Bias	Same as QC Acceptance Limits.
IS	Every field sample, standard, and QC sample. Six per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Reanalyze affected samples.	Analyst, Department Manager	Accuracy/ Bias	Same as QC Acceptance Limits.
Results between DL and LOQ	NA.	Apply "J" qualifier to results detected between DL and LOQ.	NA.	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	PCBs					
Analytical Method / SOP Reference	SW-846 8082A/ Empirical SOP-211					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix	All target analytes must be $\leq \frac{1}{2}$ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, then report sample results which are <LOQ or > 10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X LOQ.	Analyst, Department Manager	Bias/ Contamination	Same as QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD must be $\leq 30\%$ (for LCS/LCSD, if LCSD is analyzed).	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Department Manager	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be $\leq 30\%$.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager	Accuracy / Bias / Precision	Same as QC Acceptance Limits.
Surrogates	Every field and QC sample. Two per sample: Tetrachloro-m-xylene Decachlorobiphenyl	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	No corrective action will be taken when one surrogate is within criteria. If surrogates recoveries are high and sample is <LOQ, then no corrective action is taken. If surrogates recoveries are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Department Manager	Accuracy/ Bias	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	PCBs					
Analytical Method / SOP Reference	SW-846 8082A/ Empirical SOP-211					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD \leq 40%. Report the higher of the two concentrations, unless there is interference.	None. Apply "J" flag if RPD >40% and discuss in the case narrative.	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.
Results between DL and LOQ	NA.	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Department Manager	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	TAL and Waste Oil Metals (including Mercury)					
Analytical Method/SOP Reference	SW-846 6010C, 7470A, and 7471A/ Empirical SOPs 104 and 105					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples.	All target analytes must be $\leq \frac{1}{2}$ LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value; then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Department Manager	Bias/ Contamination	Same as QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%R must be within 80-120% of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Department Manager	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
Duplicate Sample	One per preparation batch of 20 or fewer samples of similar matrix.	The RPD between the original sample and duplicate should be $\leq 20\%$.	Narrate any results that are outside control limits.	Analyst, Department Manager	Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	TAL and Waste Oil Metals (including Mercury)					
Analytical Method/SOP Reference	SW-846 6010C, 7470A, and 7471A/ Empirical SOPs104 and 105					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS	One per 20 samples of similar matrix	The %R should be within 80-120%, if sample < 4x spike added.	Flag results for affected analytes for all associated samples with "N".	Analyst, Department Manager	Accuracy/Bias	Same as QC Acceptance Limits.
Serial Dilution	One per preparatory batch with sample concentration(s) >50x LOD.	The 5-fold dilution result must agree within $\pm 10\%D$ of the original sample result if result is >50x LOD.	Perform Post Digestion Spike	Analyst, Department Manager	Precision	Same as QC Acceptance Limits.
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD.	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results of samples of same matrix as estimates in SDG narrative.	Analyst, Department Manager	Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	TRPH					
Analytical Method / SOP Reference	FL-PRO / Empirical SOP338					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Must be $\leq 1/2$ the LOQ.	Re-clean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Department Manager	Bias / Contamination	Same as QC Acceptance Limits.
LCS/LCSD	One per preparation batch of 20 or fewer samples of similar matrix.	Water %Rs must be within 55-118%. Soil %Rs must be within 63-143%. If LCSD performed - The RPD between LCS and LCSD must be $\leq 20\%$ (water) and $\leq 25\%$ (soil).	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12-hour clock and acceptable, then narrate. (3) If the LCS recoveries are high but the sample results are $< LOQ$, then narrate. Otherwise prepare again and reanalyze the batch.	Analyst, Department Manager	Accuracy / Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples of similar matrix.	Water %Rs should be within 41-100%. Soil %Rs should be within 51-215%. RPD between MS and MSD should be $\leq 20\%$ (water) and $\leq 25\%$ (soil).	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable, then re-prepare the samples again and QC.	Analyst, Department Manager	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	TRPH					
Analytical Method / SOP Reference	FL-PRO / Empirical SOP338					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogates	2 per sample: 2-Fluorobiphenyl o-Terphenyl	2- Fluorobiphenyl - %Rs must meet the laboratory limits of 50-150 for waters and 50-150 for soils. o-Terphenyl - %Rs must meet the laboratory limits of 30-140 for waters and 45-135 for soils.	(1) Prepare again and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Department Manager	Accuracy /Bias	Same as 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 Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Tallahassee office.
Laboratory Documents Sample receipt, custody, and tracking record Analysis Run logs Corrective Action forms Reported field sample results Reported results for standards, QC checks, and QC samples Raw data	Laboratory documents will be included in the hardcopy and portable documents format deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Tallahassee 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 server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Pittsburgh office.
Reports SAR	All reports will be stored in hardcopy in the Tetra Tech Tallahassee project file and electronically in the server library.

Data Handling and Management - After the field investigation is completed, the field sampling log sheets will be organized by date and media and placed in the project files. The field logbooks for this project will be used only for these sites and will also be categorized and maintained in the project files after the completion of the field program. Project personnel involved in multiple field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be labeled 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 are tracked from generation to archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the subcontracted laboratory. Upon receipt of the data packages from the analytical laboratory, the Tetra Tech Project Chemist will oversee the data validation effort, which includes

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

- **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontracted laboratory are tracked in the data validation logbook. 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 Tetra Tech 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 and eventually handed over to NAVFAC.
- **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.
- **Electronic Data.** All electronic data will be compiled into a NIRIS EDD and loaded into NIRIS.

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

Matrix	Analytical Group	Sample Locations/ Identification Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory/ Organization (name and address, contact person and telephone number)
Soil, Groundwater, and Aqueous QC Samples	VOCs (plus TICs)	See Worksheet #18	SW-846 8260B	21 calendar days	Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 Brian Richard 615-345-1113 Extension 249 brichard@empirlabs.com	NA
	1,2-Dibromoethane	See Worksheet #18	SW-846 8011			
	SVOCs (including low level PAHs and TICs)	See Worksheet #18	SW-846 8270C			
	TAL and Waste Oil Metals (including mercury)	See Worksheet #18	SW-846 6010C, 7470A, and 7471A			
	PCBs	See Worksheet #18	SW-846 8082A			
	TRPH	See Worksheet #18	FL-PRO			
Groundwater	VOCs – mobile laboratory screening	See Worksheet #18	SW-846 8260B	Results within 24 hours	Analytical Laboratories of Florida 535 Riverdale Road Merritt Island, FL 32953 Dale Schamp 321-258-1355 mobilealf@cs.com	NA

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 Action (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of Corrective Action (title and organizational affiliation)
Laboratory System Audit ¹	Every two years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QA Manager or Laboratory Manager, Empirical	Laboratory QA Manager or Laboratory Manager, Empirical	Laboratory QA Manager or Laboratory Manager, Empirical
Laboratory System Audit ¹	Every year	External	FDOH	FDOH (recognized NELAP Accrediting Authority)	Laboratory QA Manager or Laboratory Manager, Empirical	Laboratory QA Manager or Laboratory Manager, Empirical	Laboratory QA Manager or Laboratory Manager, Empirical

¹ Empirical is DoD ELAP and FDOH NELAP accredited for all analytical groups and target analytes required for this project. The DoD ELAP and FDOH NELAP accreditation letters are included in Appendix D. The NELAP accreditation letter for ALF is included in Appendix C.

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	Marcia McGinnity, Laboratory QAM, Empirical	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body
Laboratory System Audit	Written audit report	Marcia McGinnity, Laboratory QAM, Empirical	Specified by NELAP	Letter	FDOH	Specified by NELAP

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 SDG	Within three weeks of receipt of laboratory data package	DVM or designee, Tetra Tech	PM and project file, Tetra Tech
Project Monthly Progress Report	Monthly for duration of project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical,	PM and project file, Tetra Tech

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

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators. See Tetra Tech SOP SA-6.3.	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, Empirical 2 - 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
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and Measurement Performance Criteria 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 not in control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
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, Empirical

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
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
	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/ 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 not in control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/ Sample Log Sheets	Ensure that sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	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	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the Measurement Performance Criteria listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		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.	
		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.	
		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 is present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36.	
IIb	SAP/ Laboratory Data Packages/ EDDs	Ensure that the LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		Discuss the impact of 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.	
		Summarize deviations from methods, procedures, or contracts in the Data Validation Report. If possible 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.	

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

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	VOCs (including TICs), 1,2-dibromoethane, SVOCs (including low level PAHs and TICs), PCBs, and TRPH	Data validation will be performed using criteria for SW-846 Methods 8260B, 8011, 8270C, 8082A, and FL-PRO listed in Worksheets #12, #15, #24, and #28, and the current DoD QSM. The logic outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review" (USEPA, October 1999) will be used to apply qualifiers to data to the extent possible.	Data Validation Specialist, Tetra Tech
IIa and IIb	Soil, Groundwater, and Aqueous QC Samples	TAL and Waste Oil Metals (including mercury)	Data validation will be performed using criteria for SW-846 Methods 6010C, 7470A, and 7471A listed in Worksheets #12, #15, #24, and #28, and the current DoD QSM. The logic outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (USEPA, October 2004) will be used to apply qualifiers to data to the extent possible.	Data Validation Specialist, Tetra Tech

Mobile laboratory VOCs data reports will not be validated.

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

DATA USABILITY ASSESSMENT

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

Completeness

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If deviations may compromise attainment of the objectives, the Tetra Tech PM 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 Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

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

Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

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 processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

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

Sensitivity

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

Project Assumptions and Data Outliers

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

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

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

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

Identify the personnel responsible for performing the usability assessment:

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

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

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

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REFERENCES

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FIGURES



DRAWN BY	DATE
K. MOORE	4/6/11
CHECKED BY	DATE
T. DECK	4/6/11
REVISED BY	DATE

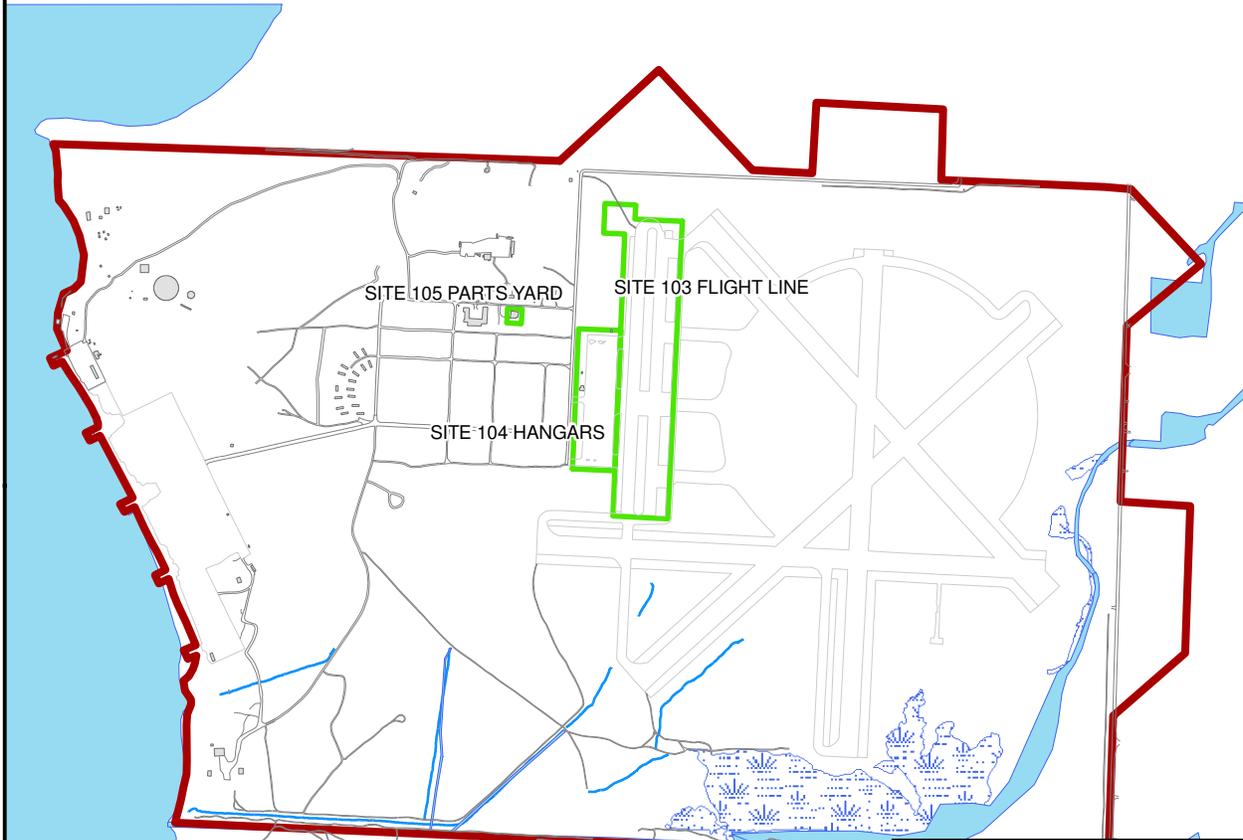
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AS NOTED



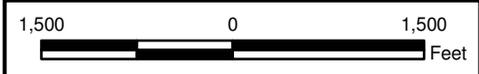
FACILITY LOCATION MAP

BRONSON FIELD
PENSACOLA, FLORIDA

CONTRACT NUMBER	CTO NUMBER
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APPROVED BY	DATE
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FIGURE ES-1	0



- Legend**
- Road
 - Airfield
 - Building
 - Site Boundary
 - Installation Boundary
 - Water
 - Stream
 - Wetland



DRAWN BY	DATE
K. MOORE	4/1/11
CHECKED BY	DATE
T. DECK	4/4/11
REVISED BY	DATE
SCALE AS NOTED	



SITE LOCATION MAP
OLF BRONSON FIELD
PENSACOLA, FLORIDA

CONTRACT NUMBER	CTO NUMBER
	JM51
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
FIGURE 10-1	0



Legend

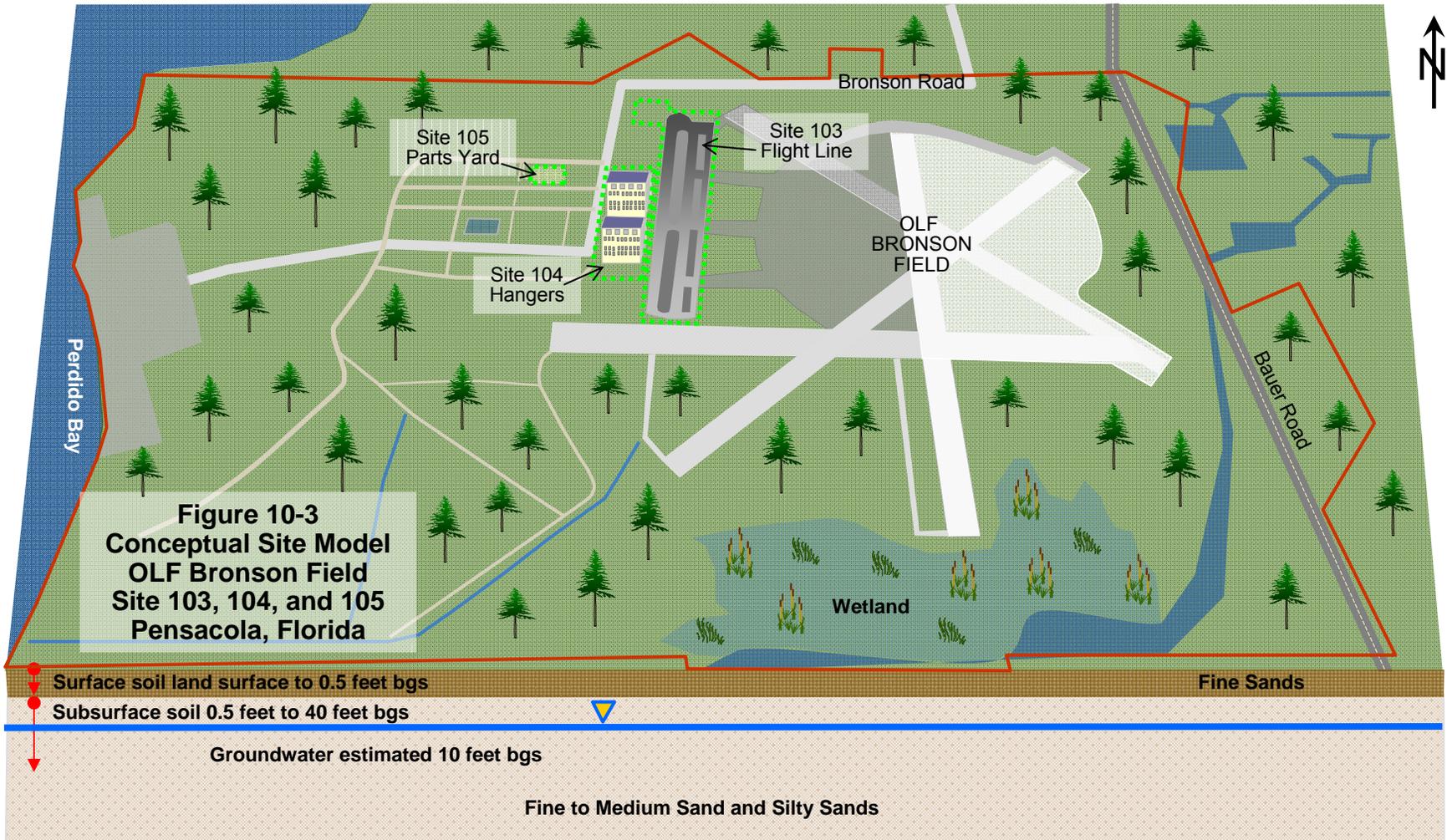
Site Boundary

DRAWN BY	DATE
K. MOORE	4/1/11
CHECKED BY	DATE
T. DECK	7/20/11
REVISED BY	DATE
SCALE AS NOTED	



SITE MAP
OLF BRONSON FIELD
PENSACOLA, FLORIDA

CONTRACT NUMBER	CTO NUMBER
_____	JM51
APPROVED BY	DATE
_____	_____
APPROVED BY	DATE
_____	_____
FIGURE NO.	REV
FIGURE 10-2	0



Potential Exposure Pathways and Receptors

Current and Future Adult and Adolescent Trespassers/ Recreational Users
Dermal contact with and incidental ingestion of soil and inhalation of dust.

Current and Future Maintenance Workers
Dermal contact with and incidental ingestion of soil and inhalation of dust.

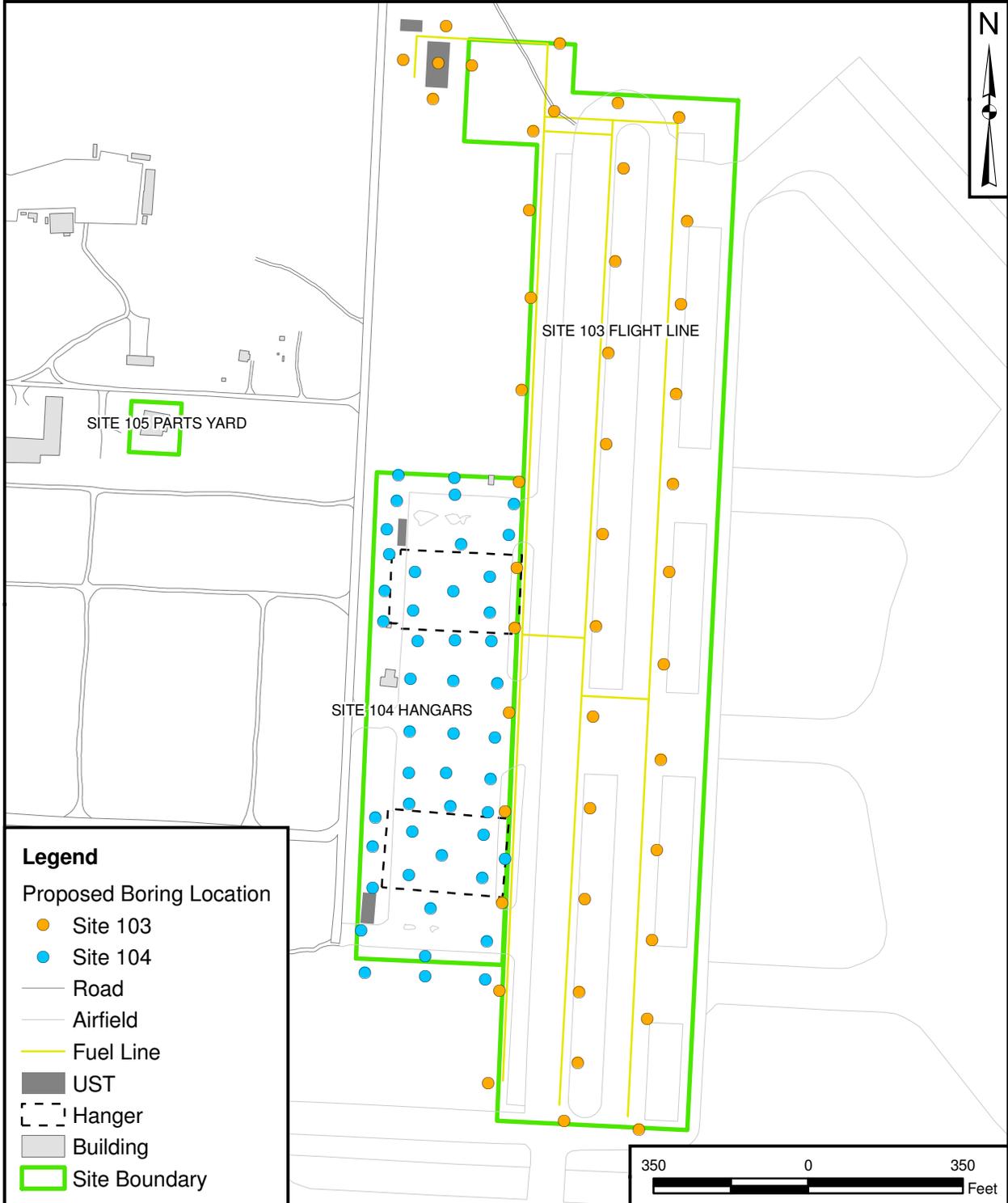
Future Construction Workers
Dermal contact with and incidental ingestion of soil and inhalation of dust.

Hypothetical Future Residents
Dermal contact with and incidental ingestion of soil and inhalation of dust.
Ingestion and inhalation of groundwater.

Note: See Section 10.4 for a description of contaminant migration pathways.

LEGEND

- Water Table
- Installation Boundary
- Stream
- Site Boundary
- OLF (Outlying Landing Field)
- below ground surface
- Native Trees
- Native Grasses
- Wetland



Legend

Proposed Boring Location

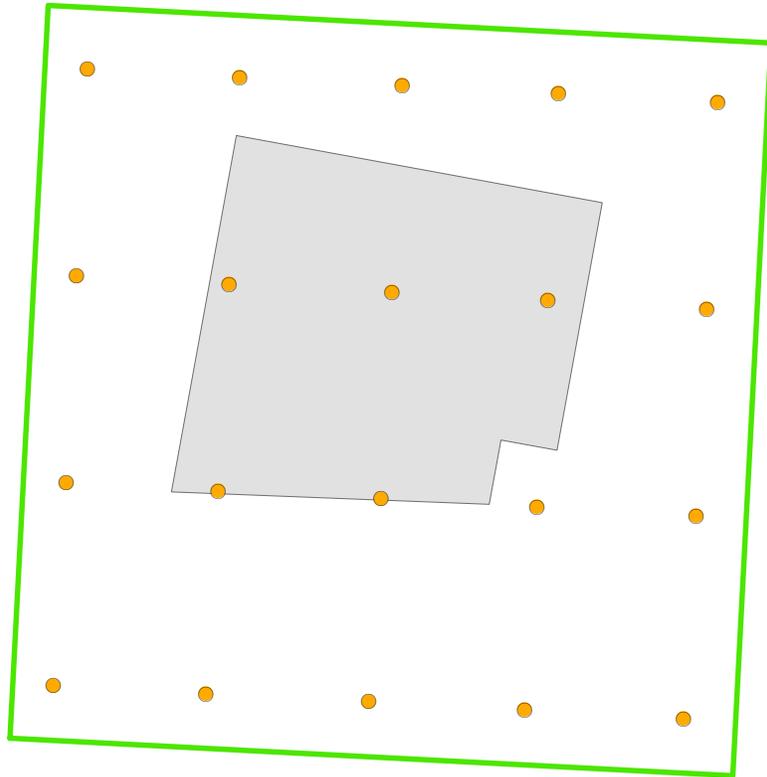
- Site 103
- Site 104
- Road
- Airfield
- Fuel Line
- UST
- - - Hanger
- Building
- Site Boundary

DRAWN BY	DATE
K. MOORE	4/1/11
CHECKED BY	DATE
T. DECK	5/25/11
REVISED BY	DATE
SCALE AS NOTED	

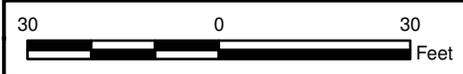


**PROPOSED SAMPLE LOCATIONS
SITES 103 AND 104
OLF BRONSON FIELD
PENSACOLA, FLORIDA**

CONTRACT NUMBER	CTO NUMBER
_____	JM51
APPROVED BY	DATE
_____	_____
APPROVED BY	DATE
_____	_____
FIGURE NO.	REV
FIGURE 17-1A	0



Legend	
●	Proposed Boring Location
—	Road
■	Building
□	Site Boundary



DRAWN BY	DATE
K. MOORE	4/1/11
CHECKED BY	DATE
T. DECK	5/25/11
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED SAMPLE LOCATIONS
SITE 105
OLF BRONSON FIELD
PENSACOLA, FLORIDA

CONTRACT NUMBER	CTO NUMBER
_____	JM51
APPROVED BY	DATE
_____	_____
APPROVED BY	DATE
_____	_____
FIGURE NO.	REV
FIGURE 17-1B	0

APPENDIX A

DATA QUALITY OBJECTIVE PRESENTATION AND MEETING MINUTES



TETRA TECH



Remedial Investigation/ Feasibility Study

Site 103 – Bronson Field Flight Line

Site 104 – Bronson Field Hangars

Site 105 – Bronson Field Parts Yard

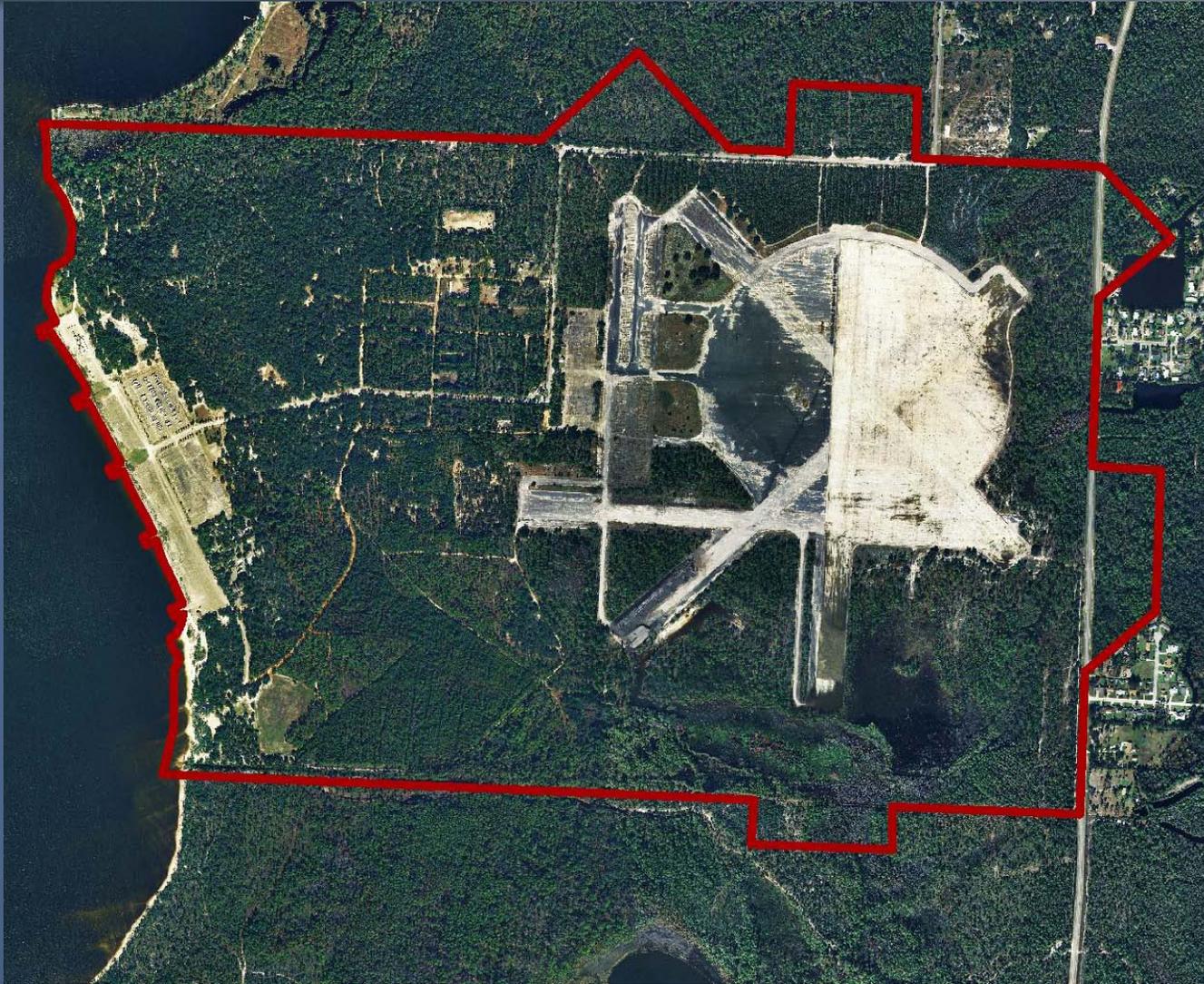
DQO Presentation

Outlying Landing Field Bronson

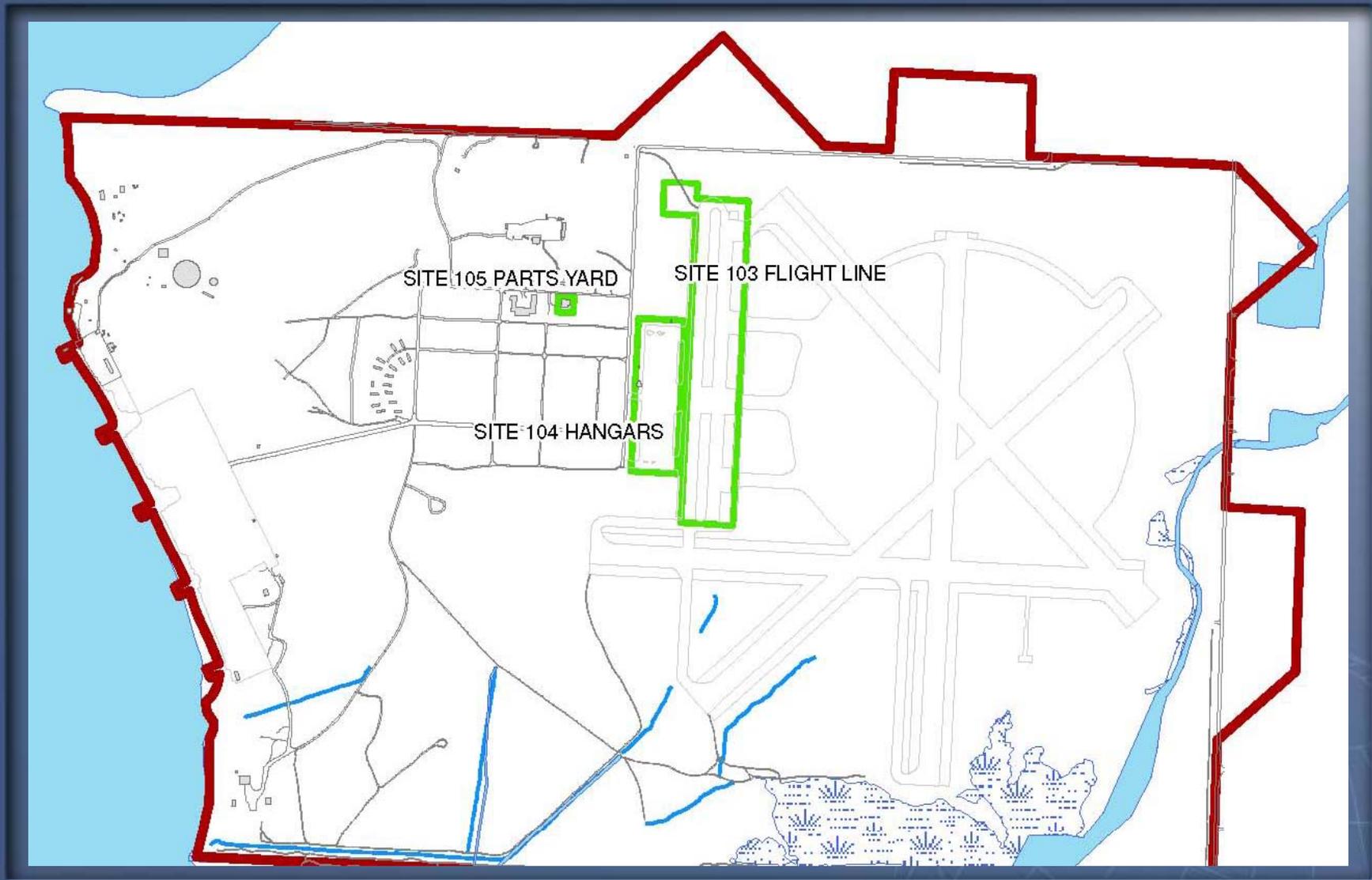
Pensacola, Florida

Contract Task Order JM51

Site Location



Site 103, 104, and 105 Location



Preliminary Assessment, 1992 - Overview

- Three areas of environmental concern were identified.
 - Fire fighting training area (Site 100), machine gun butt hill (Site 102), and aircraft fuel system (Site 103).
- Aviation gasoline (AVGAS) was used more than any other hazardous material.
- Used solvent and oil was the majority of the generated hazardous waste.
- Toluene, carbon tetrachloride, and trichloroethene were a few of the solvents used.
- 85 underground storage tanks (UST) were identified.
 - All but 35 USTs were contracted to be removed.

Preliminary Assessment, 1992 – Site 103

- Contains an aircraft fuel distribution system.
- 5 underground storage tanks (UST)
 - Four USTs - 25,000 gallon for AVGAS
 - One UST – 15,000 gallon for AVGAS
- 56 gasoline service pits for refueling airplanes
- PA noted tanks and service pits were scheduled for removal.
- 5,500 feet of gasoline fuel lines abandoned in place

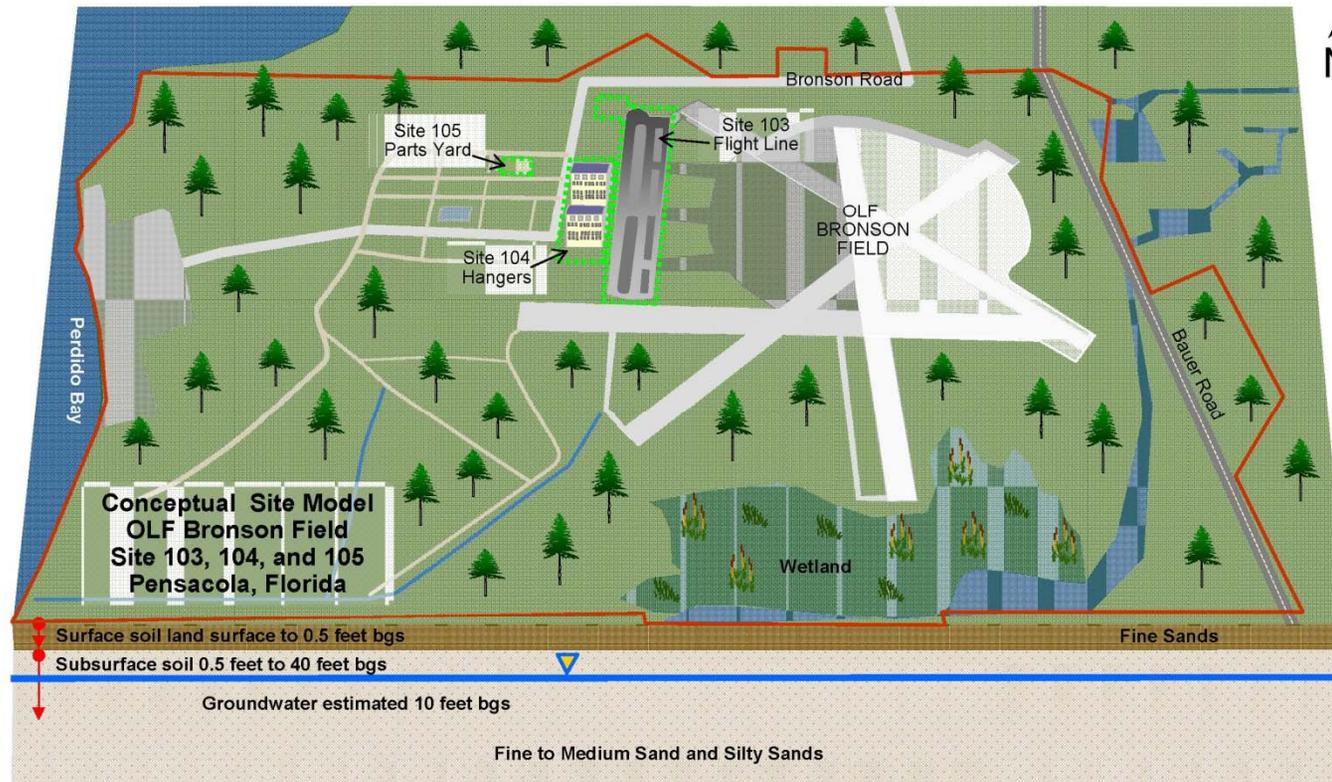
Preliminary Assessment, 1992 – Site 104

- Site 104 was not identified in PA as an area of concern.
- Former location of hangar 1103 and 1104, adjacent to Runways 9 and 18.
- Maintenance shops, kerosene tanks, lubricant oil tanks, and waste oil tanks were located at both hangars.
- Solvents, fuels, oils, and aircraft cleaners were used at and around the hangar.
- Liquid materials spilled or placed on a concrete pad may have been washed into the grass during periods of precipitation or when the pad was washed down.
- Approximately 1000 lbs of waste may have been released.

Preliminary Assessment, 1992 – Site 105

- Site 105 was not identified in PA as an area of concern.
- PA identifies tank 1156 as a 2000 gallon gasoline tank.
- 1951 figure identifies structure 1156, garage, and a battery house.
- Current aerial photos indicate structures are no longer on site.
- Currently used as storage in support of the current recreational activities.

Conceptual Site Model



Potential Exposure Pathways and Receptors

<p>Current and Future Adult and Adolescent Trespassers/ Recreational Users</p> <p>Dermal contact with and incidental ingestion of soil and inhalation of dust.</p>	<p>Current and Future Maintenance Workers</p> <p>Dermal contact with and incidental ingestion of soil and inhalation of dust.</p>	<p>Future Construction Workers</p> <p>Dermal contact with and incidental ingestion of soil and inhalation of dust.</p>	<p>Hypothetical Future Residents</p> <p>Dermal contact with and incidental ingestion of soil and inhalation of dust. Ingestion and inhalation of groundwater.</p>
---	--	---	--

LEGEND	
	Water Table
	Installation Boundry
	Stream
	Site Boundry
	Native Trees
	Native Grasses
	Wetland
	OLF Outlying Landing Field
	bgs below ground surface

Problem Statement

A Site Assessment (SA) must be conducted to determine:

- if contaminants of potential concern (COPCs) are present in surface soil, subsurface soil, and groundwater that exceed project action limits (PALs)
- the nature and extent of COPCs at Sites 103, 104, and 105

Data gathered from this investigation will be presented in the SA Report and used by the Project Team to determine the path forward for each Site.

Information Inputs

- Flame Ionization Detector (FID)/Photo Ionization Detector (PID) data
- Groundwater field parameters: dissolved oxygen, temperature, oxidation reduction potential, pH, and turbidity
- Soil and Groundwater Chemical data: TCL VOCs, TCL SVOCs, PAHs, TCL Pesticides, TCL PCBs, TAL Metals, and TRPHs
- Groundwater level measurements

Information Inputs cont'd

- Soil PALs:
 - FDEP Residential SCTLs
 - FDEP Industrial SCTLs
 - USEPA Regional Screening Levels
- Groundwater PALs:
 - FDEP GCTLs
 - USEPA Tap Water Screening Levels
 - USEPA MCLs

Study Area Boundaries - Horizontal

- The horizontal boundary is defined for each site as described below:
 - Site 103 - general vicinity of the Bronson Field Flight line and the aircraft fuel distribution system
 - Site 104 - general vicinity Bronson Field Hangars 1103 and 1104
 - Site 105 - general vicinity of Bronson Field Parts Yard
- Horizontal boundaries at Sites 103, 104 and 105 may expand based on the results of this investigation.

Study Area Boundaries - Vertical

- Groundwater
 - the shallow and deep portions of the aquifer to approximately 40 feet bls
- Soils – Site 103
 - surface soil from 0-2 ft bls
 - subsurface soil from 2 ft bls to a depth of approximately 15 feet bls
- Soils – Site 104 and 105
 - surface soil from 0-0.5 ft bls
 - surface soil from 0.5-2 ft bls
 - subsurface soil from 2 ft bls to a depth of approximately 15 feet bls

Analytic Approach: COPC Decision Rule

- For each target analyte in each investigated medium, if the maximum measured chemical concentration does not exceed its PAL, then exclude the chemical from further consideration.
- For each target analyte in each investigated medium, if the maximum measured chemical concentration exceeds its PAL, then retain the chemical as a COPC.

Analytic Approach: Delineation Decision Rule

- If the measured surface soil, subsurface soil, and groundwater chemical concentrations are sufficient to delineate the extent of contamination in those media, then stop collecting data.
- If the data are not sufficient to determine the extent of surface soil, subsurface soil, and groundwater contamination, then conduct another phase of field sampling to delineate COPCs in each medium.

Analytic Approach: Risk Based Corrective Action

Once the COPCs have been identified and delineated and the investigation is complete, the data will be evaluated in accordance with FDEP 62-780, F.A.C. Risk Based Corrective Action (RBCA) process to determine an appropriate Risk Management Option (RMO).

- Groundwater at Sites 103, 104, and 105
 - If the maximum measured chemical concentration of a COPC is less than its GCTL then proceed to NFA without Institutional Controls. Otherwise the project team will consider risk to site personnel and remedial alternatives in a RAP.

Analytic Approach: Risk Based Corrective Action

- Soils at Site 103 and 104
 - Decision Rule #1
 - If the maximum measured chemical concentration of a COPC is less than its Residential SCTL then proceed to NFA without Institutional Controls. Otherwise proceed to Rule #2.
 - Decision Rule #2
 - If the maximum measured chemical concentration of a COPC is less than its Industrial SCTL then proceed to NFA with Institutional Controls. Otherwise the project team will consider risk to site personnel and remedial alternatives in a RAP.

Analytic Approach: Risk Based Corrective Action

- Soils at Site 105
 - Decision Rule #1
 - If a COPCs 95% UCL concentration is less than its Residential SCTL and soil concentrations are less than the leachability-based SCTL, then proceed to NFA without Institutional controls. Otherwise proceed to Rule #2
 - Decision Rule #2
 - If a COPCs 95% UCL concentration is less than its Industrial SCTL then proceed to NFA with Institutional controls. Otherwise the project team will consider risk to site personnel and remedial alternatives in a RAP.

Data Collection Plan: DPT Investigation

Field screening methods will be used during the first phase of sampling to determine the initial vertical and horizontal extent of potential contamination in groundwater and soil.

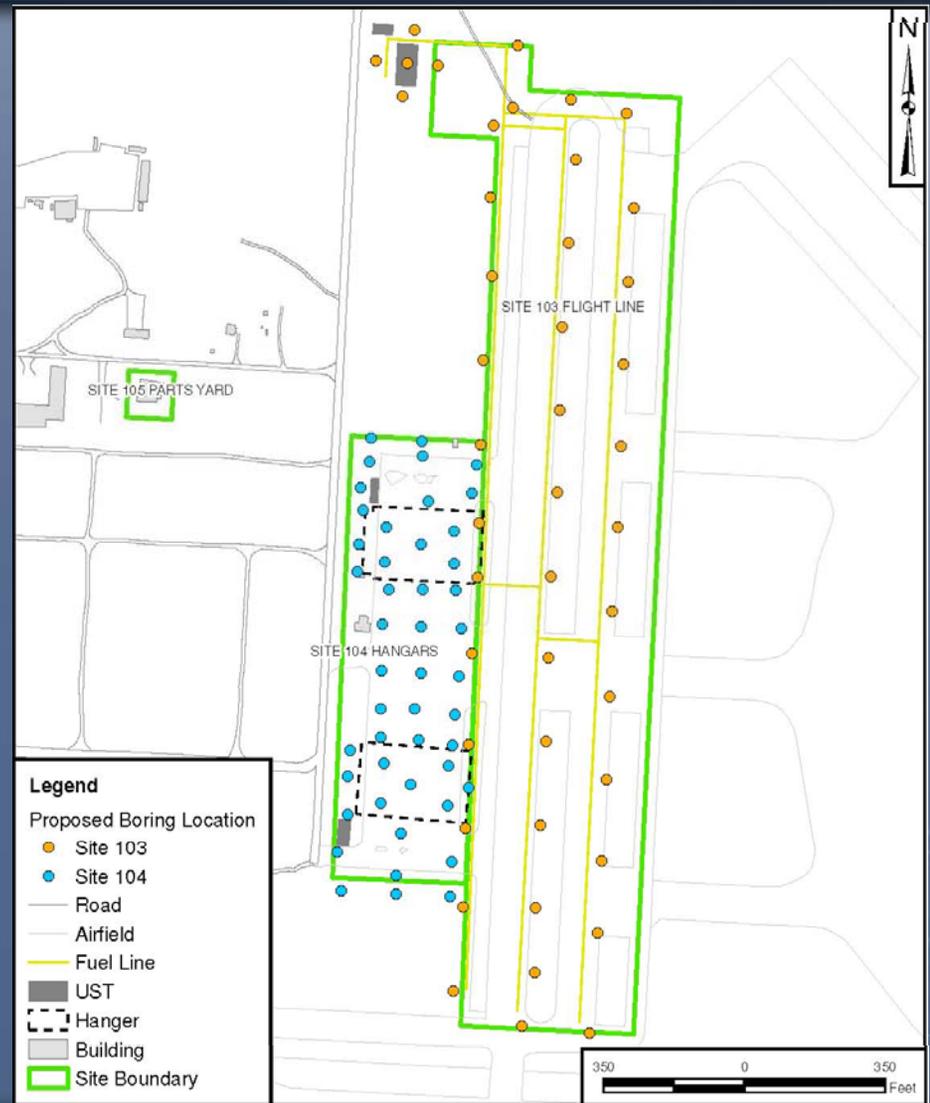
- DPT soil and groundwater samples will be collected.
- Soils will be screened with a FID/PID and visual inspection (oily residue). Two samples will be collected from each interval based on screening

Data Collection Plan: DPT Investigation cont'd

- Site 103 Soil samples will be analyzed for VOCs, SVOCs (including PAHs), waste oil group metals, and TRPH.
- Site 104 and 105 soil samples will be analyzed for VOCs, SVOCs (including PAHs), Pesticides, PCBs, metals, and TRPH.
- Groundwater samples will be analyzed for VOCs by an on-site mobile laboratory (10% to fixed based lab for confirmation)

Proposed Sample Locations

- Site 103 – 44 borings
 - 88 soil samples
 - 44 groundwater samples
- Site 104 – 48 borings
 - 96 soil samples
 - 48 groundwater samples
- Total of 9 groundwater samples to a fixed based lab.



Proposed Sample Locations cont'd

- Site 105 – 20 boring
 - 40 soil samples
 - 20 groundwater samples
- Total of 2 groundwater samples to a fixed based lab.



Analytic Approach – Permanent Monitoring Wells

Based on the results of the DPT groundwater investigation, new monitoring wells will be installed during a second phase of sampling.

- New permanent monitoring wells
 - Site 103 – 22 shallow and 3 deep monitoring wells
 - Site 104 – 24 shallow and 3 deep monitoring wells
 - Site 105 – 10 shallow and 3 deep monitoring wells

Analytic Approach – Permanent Monitoring Wells cont'd

- Site 103 groundwater samples will be analyzed for VOCs, SVOCs (including PAHs), waste oil group metals, and TRPH.
- Site 104 and 105 groundwater samples will be analyzed for VOCs, SVOCs (including PAHs), Pesticides, PCBs, metals, and TRPH.

Questions?





Bronson Field DQO Meeting

May 19, 2011

Date: May 19, 2011

Time: 10:30 am

Call-in Number and code: 866/692-5721 1802299#

Purpose: This teleconference was held to discuss DQO at Bronson Field

Participants:

Patty Marajh-Whittemore, NAVFAC SE
Ken Bowers, NAVFAC -Atlantic
Greg Campbell, NAS PWD
David Grabka, FDEP
Gerry Walker, Tetra Tech
Frank Lesesne, Tetra Tech
Amber Igoe, Tetra Tech
Peggy Churchill, Tetra Tech
Tom Deck, Tetra Tech
Kelly Carper, Tetra Tech

PowerPoint Presentation

Background Section presented by Tom Deck

Q: What is the box located at the north end of the flight line?

A: PA identified it as USTs.

Q: Is the machine gun butt area part of the MMRP site?

A: It was a CERCLA RI Site, but has received a NFA.

Q: Is there a Site 101?

A: No, there was no Site identified as 101 in the PA.

Q: Did the gasoline service pits use AVGAS?

A: Yes

Q: Does roundwater tends to flow to Perdido Bay?

A: Overall it tends to flow towards Perdido Bay; however there are some localized drainage features to the South.

Q: Where are the possible releases?

A: Releases could have occurred at the USTs, the bowsers, along the pipeline and from historic activities at the hangars. Site 105 also has potential releases from the former gasoline tank and battery storage. The assumption is groundwater is approximately 10 feet below land surface. Exposure standards will be based on FDEP's 62-770 and 62-780 guidance and exposure



scenarios will be: current and future trespassers, current and future maintenance workers, future construction and future residential.

Q: What type of recreational activities are currently ongoing at Bronson?

A: The moral and welfare group is currently housed at the facility, there is Frisbee golf area, the field is used for model aircraft enthusiasts and the Santa Rosa County Sheriff's Department uses the area for training.

Q: Is the pipeline still present?

A: Yes and the bowser as intact as well.

Q: Is Site 105 being used for equipment storage?

A: Yes, the morale and welfare group currently stores equipment and historically it was a garage and battery storage area. The Santa Rosa County Sheriff's Department also currently has a storage area at Site 104.

Q: What are the historic use timeframes for the three sites?

A: The base was established in 1942 and closed shortly after WWII.

Problem Statement Section presented by Peggy Churchill

Q: Are the full suite of constituents being run at all 3 Sites?

A: The following list of constituents was developed for the 3 Sites

Site 103 Flight line

--TCL VOCs (TICs included), TCL sVOCs (PAHs and TICs included), TCL PCBs, waste oil group metals (Table C 62-770) and TRPH. For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (eg PCBs) or reduced (e.g metals) for groundwater sampling event(s).

Site 104

--TCL VOCs (TICs included), TCL sVOCs (PAHs and TICs included), TCL PCBs, TAL metals and TRPH. As long as the hangars weren't used for mosquito control or there was evidence in the PA pesticides were stored in the hangars, pesticides can be removed from the analyte list. The TAL metals will be analyzed during the first round; if any of metals from the TAL list are not-detected then the list can be reduced. For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (eg PCBs) or reduced (e.g metals) for groundwater sampling event(s).

Site 105

--TCL VOCs (TICs included), TCL sVOCs (PAHs and TICs included), TCL PCBs, TAL metals and TRPH. The TAL metals will be analyzed during the first round; if any of metals from the TAL list are not-detected then the list can be reduced. For the time being, the same analyte list will be used for both soil and groundwater; the soil data will be reviewed to see if analytes can be eliminated (eg PCBs) or reduced (e.g metals) for groundwater sampling event(s).

Slide 11:

--FDEPs leachability to groundwater will be added to the list.



Memorandum

Slide 14: Decision Rule:

--The statement "If analytes are not above SCTLs or leachability to groundwater in soil they will not be analyzed in groundwater" will be added.

Slide 15:

--Delineation will be removed from the decision rule at this time. The vertical and horizontal component will be listed as an objective.

Slide 16:

--If there are no exceedances for groundwater, then NFA will be recommended
--The use of "A COC" will be changed to all target analytes

Slide 17:

--FDEP regulations for RMOs will be referred to in the UFP-SAP.

Data Collection Plan presented by Frank Lesesne

--It was suggested that Site 103 will also need OVA headspace in addition to the PID/FID screening

Slide 19

Q: When collecting groundwater samples with the DPT, do we need to send 10% of the samples (9 samples) to fixed based lab for confirmation?

A: Yes. The mobile lab will be analyzing DPT screening the groundwater samples for volatiles only, a determination will be made on the analyte list for the confirmation samples that will be sent to the fixed base laboratory.

Slide 20:

Q: Do we need to get a variance to collect samples every 200 feet instead of every 20 feet?

A: It will depend on whether or not the tanks/lines have been properly closed and if inspectors have signed off on the closure. Based on the current information, we would probably need to perform closure using an alternate procedure. David Grabka will speak with the petroleum group at FDEP.

Questions/End of Meeting Discussion:

--The decision for the well placement will be decided after the soil and DPT groundwater screening data is obtained; wells can be shifted from one Site to another if necessary and the well depths are subject to change. The Partnering Team will make the determination collectively after reviewing all of the analytical data.

--Site tour scheduled for May 23rd 2011 3:30 (CST).

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES AND FIELD FORMS



TETRA TECH NUS,
INC.

STANDARD OPERATING PROCEDURES

Number	CT-04	Page	1 of 7
Effective Date	03/09/09	Revision	2
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston		

Subject
SAMPLE NOMENCLATURE

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Subject SAMPLE NOMENCLATURE	Number CT-04	Page 2 of 7
	Revision 2	Effective Date 03/09/09

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 3 of 7
	Revision 2	Effective Date 03/09/09

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alphanumeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

- (1) Soil and sediment sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	NNNN 4 Characters
Site identifier	Sample type	Sample location	Sample depth

- (2) Aqueous (groundwater or surface water) sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	NN 2 Characters	-A 1 Character
Site identifier	Sample type	Sample location	Round number	Filtered sample only

- (3) Biota sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	AA 2 Characters	NNN 3 Characters
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 4 of 7
	Revision 2	Effective Date 03/09/09

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

- A01 - Area of Concern (AOC) 1
- 125 - SWMU 125
- 000 - Base- or facility-wide sample (e.g., upgradient well)
- BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

- AH - Ash Sample

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 5 of 7
	Revision 2	Effective Date 03/09/09

- AS - Air Sample
- BM - Building Material Sample
- BSB - Biota Sample Full Body
- BSF - Biota Sample Fillet
- CP - Composite Sample
- CS - Chip Sample
- DS - Drum Sample
- DU - Dust Sample
- FP - Free Product
- IDW - Investigation-Derived Waste Sample
- LT - Leachate Sample
- MW - Monitoring Well Groundwater Sample
- OF - Outfall Sample
- RW - Residential Well Sample
- SB - Soil Boring Sample
- SD - Sediment Sample
- SC - Scrape Sample
- SG - Soil Gas Sample
- SL - Sludge Sample
- SP - Seep Sample
- SS - Surface Soil Sample
- ST - Storm Sewer Water Sample
- SW - Surface Water Sample
- TP - Test Pit Sample
- TW - Temporary Well Sample
- WC - Well Construction Material Sample
- WP - Wipe Sample
- WS - Waste/Solid Sample
- WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

- 001 - Monitoring well 1
- N32E92 - Grid location 32 North and 92 East
- D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

- BC - Blue Crab
- GB - Blue Gill
- CO - Corn
- SB - Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 6 of 7
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A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

- TB = Trip Blank
- RB = Rinsate Blank (Equipment Blank)
- FD = Field Duplicate
- AB = Ambient Conditions Blank
- WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

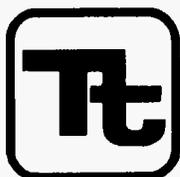
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The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Applicability Tetra Tech NUS, Inc.	
Prepared Management Information Systems Department	
Approved D. Senovich <i>[Signature]</i>	

Subject
DATABASE RECORDS AND QUALITY ASSURANCE

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

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Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request Form included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

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Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

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5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of-Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

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editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: _____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

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. _____	

FC 1000. CLEANING / DECONTAMINATION PROCEDURES

1. PERFORMANCE CRITERIA

- 1.1. The cleaning/decontamination procedures must ensure that all equipment that contacts a sample during sample collection is free from the analytes of interest and constituents that would interfere with the analytes of interest.
- 1.2. The detergents and other cleaning supplies cannot contribute analytes of interest or interfering constituents unless these are effectively removed during a subsequent step in the cleaning procedure.
- 1.3. The effectiveness of any cleaning procedure (including all cleaning reagents) must be supported by equipment blanks with reported non-detected values.

The cleaning procedures outlined in this SOP are designed to meet the above-mentioned performance criteria. Alternative cleaning reagents or procedures may be used. However, the organization must be prepared to demonstrate through documentation (i.e., company-written protocols and analytical records) and historical data (i.e., absence of analytes of interest in equipment blanks) that it consistently meets these performance criteria. Field quality control measures (see FQ 1210) must support the use of alternative reagents or procedures.

FC 1001. *Cleaning Reagents*

Recommendations for the types and grades of various cleaning supplies are outlined below. The recommended reagent types or grades were selected to ensure that the cleaned equipment is free from any detectable contamination.

1. DETERGENTS: Use Luminox (or a non-phosphate solvent based equivalent), Liqui-Nox (or a non-phosphate equivalent) or Alconox (or equivalent). EPA recommends Luminox (or equivalent) since solvent rinses can be eliminated from the cleaning process. Liquinox (or equivalent) may be substituted (solvent rinses, when applicable, must be performed), and Alconox (or equivalent) may be substituted if the sampling equipment will not be used to collect phosphorus or phosphorus-containing compounds.
2. SOLVENTS

Note: If the detergent Luminox (or equivalent) is used, solvent rinses are not required.

- 2.1. Use pesticide grade isopropanol as the rinse solvent in routine equipment cleaning procedures. This grade of alcohol must be purchased from a laboratory supply vendor.
- 2.2. Other solvents, such as acetone or methanol, may be used as the final rinse solvent if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics.
 - 2.2.1. **Do not use** acetone if volatile organics are of interest.
- 2.3. Properly dispose of all wastes according to applicable regulations. Containerize all solvents (including rinsates) for on-site remediation or off-site disposal, as required.
- 2.4. Pre-clean equipment that is heavily contaminated (see FC 1120, section 3) with organic analytes with reagent grade acetone and hexane or other suitable solvents.
- 2.5. Use pesticide grade methylene chloride when cleaning sample containers.

2.6. Store all solvents away from potential sources of contamination (gas, copier supplies, etc.).

3. ANALYTE-FREE WATER SOURCES

3.1. Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits.

3.2. Maintain documentation (such as results from equipment blanks) to demonstrate the reliability and purity of analyte-free water source(s).

3.3. The source of the water must meet the requirements of the analytical method and must be free from the analytes of interest. In general, the following water types are associated with specific analyte groups:

- Milli-Q (or equivalent polished water): suitable for all analyses.
- Organic-free: suitable for volatile and extractable organics.
- Deionized water: not suitable for volatile and extractable organics if the analytes of interest are present in concentrations that affect the result.
- Distilled water: not suitable for volatile and extractable organics, metals or ultra-trace metals.

3.4. Use analyte-free water for blank preparation and the final decontamination water rinse.

3.5. In order to minimize long-term storage and potential leaching problems, obtain or purchase analyte-free water just prior to the sampling event. If obtained from a source (such as a laboratory), fill the transport containers and use the contents for a single sampling event. Empty the transport container(s) at the end of the sampling event.

3.6. Discard any analyte-free water that is transferred to a dispensing container (such as a wash bottle) at the end of each sampling day.

4. ACIDS

4.1. Reagent Grade Nitric Acid: 10 - 15% (one volume concentrated nitric acid and five volumes deionized water).

4.1.1. Use for the acid rinse unless nitrogen components (e.g., nitrate, nitrite, etc.) are to be sampled.

4.1.2. If sampling for ultra-trace levels of metals, use an ultra-pure grade acid.

4.2. Reagent Grade Hydrochloric Acid: 10% hydrochloric acid (one volume concentrated hydrochloric and three volumes deionized water).

4.2.1. Use when nitrogen components are to be sampled.

4.3. If samples for both metals and the nitrogen-containing components (see FC 1001, section 4.1.1 above) are collected with the equipment, use the hydrochloric acid rinse, or thoroughly rinse with hydrochloric acid after a nitric acid rinse.

4.4. If sampling for ultra trace levels of metals, use an ultra-pure grade acid.

4.5. Freshly prepared acid solutions may be recycled during the sampling event or cleaning process. Dispose appropriately at the end of the sampling event, cleaning process or if acid is discolored or appears otherwise contaminated (e.g., floating particulates).

4.5.1. Transport only the quantity necessary to complete the sampling event.

- 4.6. Dispose of any unused acids according to FDEP and local ordinances.

FC 1002. *Reagent Storage Containers*

The contents of all containers must be clearly marked.

1. DETERGENTS: Store in the original container or in a high density polyethylene (HDPE) or polypropylene (PP) container.
2. SOLVENTS
 - 2.1. Store solvents to be used for cleaning or decontamination in the original container until use in the field. If transferred to another container for field use, the container must be either glass or Teflon.
 - 2.2. Use dispensing containers constructed of glass, Teflon, or stainless steel. Note: if stainless steel sprayers are used, any components (including gaskets and transfer lines) that contact the solvents must be constructed of inert materials.
3. ANALYTE-FREE WATER: Transport in containers appropriate to the type of water to be stored. If the water is commercially purchased (e.g., grocery store), use the original containers when transporting the water to the field. Containers made of glass, Teflon, polypropylene, or Polyethylene (PE) are acceptable.
 - 3.1. Use glass, Teflon, polypropylene or PE to transport organic-free sources of water on-site.
 - 3.2. Dispense water from containers made of glass, Teflon, PE or polypropylene.
 - 3.3. Do not store water in transport containers for more than three days before beginning a sampling event.
 - 3.4. Store and dispense acids using containers made of glass, Teflon, PE or polypropylene.

FC 1003. *General Requirements*

1. Before using any equipment, clean/decontaminate all sampling equipment (pumps, tubing, lanyards, split spoons, etc.) that are exposed to the sample.
 - 1.1. Before installing, clean (or obtain as certified precleaned) all equipment that is dedicated to a single sampling point and remains in contact with the sample medium (e.g., permanently installed groundwater pump (see FS 2220, section 3.3.4)).
 - 1.2. Clean this equipment any time it is removed for maintenance or repair.
 - 1.3. Replace dedicated tubing if discolored or damaged.
2. Clean all equipment in a designated area having a controlled environment (house, laboratory, or base of field operations) and transport to the field precleaned and ready to use, unless otherwise justified.
3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.
4. Whenever possible, transport sufficient clean equipment to the field so that an entire sampling event can be conducted without the need for cleaning equipment in the field.

5. Segregate equipment that is only used once (i.e., not cleaned in the field) from clean equipment and return to the in-house cleaning facility to be cleaned in a controlled environment.
6. Protect decontaminated field equipment (including well sounders) from environmental contamination by securely wrapping and sealing with one of the following:
 - 6.1. Aluminum foil (commercial grade is acceptable);
 - 6.2. Untreated butcher paper; or
 - 6.3. Clean, untreated, disposable plastic bags. Plastic bags may be used:
 - For all analyte groups except volatile and extractable organics;
 - For volatile and extractable organics, if the equipment is first wrapped in foil or butcher paper or if the equipment is completely dry.
7. Containerize all solvent rinsing wastes, detergent wastes and other chemical wastes requiring off-site or regulated disposal. Dispose of all wastes in conformance with applicable regulations.

FC 1100. Cleaning Sample Collection Equipment

FC 1110. ON-SITE/IN-FIELD CLEANING

1. Cleaning equipment on-site is not recommended because:
 - 1.1. Environmental conditions cannot be controlled.
 - 1.2. Wastes (solvents and acids) must be containerized for proper disposal.
2. If performed, follow the appropriate cleaning procedure as outlined in FC 1130. Ambient temperature water may be substituted in the hot, sudsy water bath, and hot water rinses.

Note: Properly dispose of all solvents and acids.

3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.

FC 1120. HEAVILY CONTAMINATED EQUIPMENT

In order to avoid contaminating other samples, isolate heavily contaminated equipment from other equipment and thoroughly decontaminate the equipment before further use. Equipment is considered heavily contaminated if it:

- Has been used to collect samples from a source known to contain significantly higher levels than background;
 - Has been used to collect free product; or
 - Has been used to collect industrial products (e.g., pesticides or solvents) or their by-products.
1. Cleaning heavily contaminated equipment in the field is not recommended.
 2. ON-SITE PROCEDURES
 - 2.1. Protect all other equipment, personnel and samples from exposure by isolating the equipment immediately after use.

- 2.2. At a minimum, place the equipment in a tightly sealed untreated plastic bag.
 - 2.3. Do not store or ship the contaminated equipment next to clean, decontaminated equipment, unused sample containers, or filled sample containers.
 - 2.4. Transport the equipment back to the base of operations for thorough decontamination.
 - 2.5. If cleaning must occur in the field, and in order to document the effectiveness of the procedure, collect and analyze blanks on the cleaned equipment (see FQ 1000).
3. CLEANING PROCEDURES
- 3.1. If organic contamination cannot be readily removed with scrubbing and a detergent solution, prerinse equipment by thoroughly rinsing or soaking the equipment in acetone.
 - 3.1.1. Do not use solvent soaks or rinses if the material is clear acrylic.
 - 3.1.2. Use hexane only if preceded and followed by acetone.
 - 3.2. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with routine cleaning procedures.
 - 3.3. After the solvent rinses (and/or steam cleaning), use the appropriate cleaning procedure (see FC 1130).
 - 3.3.1. Scrub, rather than soak all equipment with sudsy water.
 - 3.3.2. If high levels of metals are suspected and the equipment cannot be cleaned without acid rinsing, soak the equipment in the appropriate acid. Do not use stainless steel equipment when heavy metal contamination is suspected or present, since stainless steel cannot be exposed to prolonged acid soaks.
 - 3.4. If the field equipment cannot be cleaned utilizing these procedures, discard unless further cleaning with stronger solvents and/or oxidizing solutions is effective as evidenced by visual observation and blanks.
 - 3.5. Clearly mark or disable all discarded equipment to discourage use.

FC 1130. GENERAL CLEANING

Follow these procedures when cleaning equipment under controlled conditions. See FC 1110 for modifications if cleaning is performed on-site. Check manufacturer's instructions for cleaning restrictions and/or recommendations.

FC 1131. Procedure for Teflon, Stainless Steel and Glass Sampling Equipment

This procedure must be used when sampling for **ALL** analyte groups: extractable organics, metals, nutrients, etc. or if a single decontamination protocol is desired to clean all Teflon, stainless steel and glass equipment.

1. Rinse equipment with hot tap water.
2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).
3. If necessary, use a brush to remove particulate matter or surface film.
4. Rinse thoroughly with hot tap water.

5. If samples for trace metals or inorganic analytes will be collected with the equipment and the equipment **is not** stainless steel, thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4).
6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water.
7. If samples for volatile or extractable organics will be collected, rinse with isopropanol. Wet equipment surfaces thoroughly with free-flowing solvent. Rinse thoroughly with analyte-free water (see FC 1001, section 3).
8. Allow to air dry. Wrap and seal according to FC 1003, section 6 as soon as the equipment is air-dried.
9. If isopropanol is used, the equipment may be air-dried without the final analyte-free water rinse (see FC 1131, section 8 above); however, **the equipment must be completely dry before wrapping or use.**
10. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1132. *General Cleaning Procedure for Plastic Sampling Equipment*

1. Rinse equipment with hot tap water.
2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).
3. If necessary, use a brush to remove particulate matter or surface film.
4. Rinse thoroughly with hot tap water.
5. Thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4).
6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water. Allow to air dry as long as possible.
7. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1133. *Cleaning Procedure by Analyte Group*

See Table FC 1000-1 for the procedures to be used to decontaminate equipment based on construction of sampling equipment, and analyte groups to be sampled.

~~**FC 1140.** **AUTOMATIC SAMPLERS, SAMPLING TRAINS AND BOTTLES**~~

- ~~1. When automatic samplers are deployed TD
7/20/11 ed time periods, clean the sampler using the following procedures when routine maintenance is performed. Inspect deployed samplers prior to each use. At a minimum, change the tubing if it has become discolored or has lost elasticity (FC 1140, section 2.3 below).~~
- ~~2. Clean all automatic samplers (such as ISCO) as follows:
 - ~~2.1. Wash the exterior and accessible interior portions of the automatic samplers (excluding the waterproof timing mechanisms) with laboratory detergent (see FC 1001, section 1) and rinse with tap water.~~~~

- 2.2. Clean the face of the timing case mechanisms with a clean, damp cloth.
- 2.3. Check all tubing (sample intake and pump tubing). Change the tubing every six months (if used frequently) or if it has become discolored (i.e., affected by mold and algae) or if it has lost its elasticity.
- 2.4. See FC 1160, section 4 for the procedures associated with cleaning the tubing in the pump head.
3. AUTOMATIC SAMPLER ROTARY FUNNEL AND DISTRIBUTOR
 - 3.1. Clean with hot sudsy water and a brush (see FC 1001, section 1 for appropriate detergent type).
 - 3.2. Rinse thoroughly with analyte-free water.
 - 3.3. Air dry.
 - 3.4. Replace in sampler.
4. SAMPLER METAL TUBE: Clean as outlined in FC 1160, section 5.
5. REUSABLE GLASS COMPOSITE SAMPLE CONTAINERS
 - 5.1. If containers are used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the container several times with reagent-grade acetone before the detergent wash. If material cannot be removed with acetone, discard the container.
 - 5.2. Wash containers following the procedure outlined in FC 1131 above. End with a final solvent rinse if organics are to be sampled.
 - 5.3. Invert containers to drain and air dry for at least 24 hours.
 - 5.4. Cap with aluminum foil, Teflon film or the decontaminated Teflon-lined lid.
 - 5.5. After use, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet, and return to the laboratory or base of operations.
 - 5.6. **Do not recycle or reuse containers if:**
 - 5.6.1. They were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;
 - 5.6.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or
 - 5.6.3. The containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers must be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.
 - 5.6.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest **before** use. If found to be contaminated, (i.e., constituents of interest are found at method detection levels or higher), then **discard the containers.**
6. REUSABLE PLASTIC COMPOSITE SAMPLE CONTAINERS
 - 6.1. Follow FC 1132.

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- 6.2. Inspect the containers. Determine if the containers can be reused by the criteria in FC 1140, section 5 above.
7. GLASS SEQUENTIAL SAMPLE BOTTLES FOR AUTOMATIC SAMPLER BASED FOR SEQUENTIAL MODE
- 7.1. Clean glass sequential sample bottles to be used for collecting inorganic samples by using a laboratory dishwasher (see FC 1140, sections 7.1.1 through 7.1.3 below) or manually following the procedures in FC 1131.
- 7.1.1. Rinse with appropriate acid solution (see FC 1001, section 4).
- 7.1.2. Rinse thoroughly with tap water.
- 7.1.3. Wash in dishwasher a TD
7/20/11 le, using laboratory detergent cycle, followed by tap and analyte-free water rinse.
- 7.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.
- 7.3. Rinse bottles in the field with water as soon as possible after sampling event.
8. Glass Sequential Sample Bottles (Automatic Sampler based for Sequential Mode) to be used for Collecting Samples for Organic Compounds
- 8.1. Use cleaning procedures outlined in FC 1131. Allow containers to thoroughly air dry before use.
- 8.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.
9. BOTTLE SIPHONS USED TO TRANSFER SAMPLES FROM COMPOSITE CONTAINERS
- 9.1. Rinse tubing with solvent and dry overnight in a drying oven.
- 9.2. Cap ends with aluminum foil and/or Teflon film for storage.
- 9.3. Seal in plastic for storage and transport.
- 9.4. Flush siphon thoroughly with sample before use.
10. REUSABLE TEFLON COMPOSITE MIXER RODS
- 10.1. Follow procedures outlined in FC 1131.
- 10.2. Wrap in aluminum foil for storage.

FC 1150. FILTRATION EQUIPMENT

1. Dissolved Constituents using in-line, Molded and Disposable Filter Units
- 1.1. Peristaltic Pump
- 1.1.1. Clean the pump following procedures in FC 1170, section 2.2.
- 1.1.2. Clean the pump head tubing following FC 1160, section 4.
- 1.1.3. If Teflon tubing is used, clean following the procedures in FC 1160, section 3.
- 1.1.4. Clean other tubing types such as polyethylene according to the appropriate procedures listed in FC 1160, section 7.
- 1.2. Other Equipment Types (e.g., pressurized Teflon bailer)

- 1.2.1. Follow the appropriate cleaning regimen specified in FC 1131 through FC 1132 for other types of equipment including in-line, molded and disposable filters.
2. Dissolved Constituents using Nitratable Filtration Units (e.g., syringes, "tripod assembly")
- 2.1. Stainless Steel or Glass Units
- 2.1.1. Follow FC 1131, assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.
- 2.1.2. Remove and clean any transfer tubing according to the appropriate cleaning procedures (see FC 1160).
- 2.1.3. Assemble the unit and cap both the pressure inlet and sample discharge lines (or whole unit if a syringe) with aluminum foil to prevent contamination during storage.
- 2.1.4. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.
- 2.2. Reusable In-Line Filter Holders
- 2.2.1. Clean, using FC 1131, (if Teflon, glass or stainless steel) or FC 1132 (if plastic) assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.
- 2.2.2. Assemble the unit and wrap with aluminum foil to prevent contamination during storage.
- 2.2.3. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.
3. FILTERS
- 3.1. Do not clean filters. Instructions for rinsing the filters prior to use are discussed in the applicable sampling SOPs (FS 2000 - FS 8000).

FC 1160. SAMPLE TUBING DECONTAMINATION

1. Check tubing:
- 1.1. For discoloration: Remove discolored tubing from use until it can be cleaned. If the discoloration cannot be removed, discard the tubing.
- 1.2. For elasticity (if used in a peristaltic-type pump): Discard any tubing that has lost its elasticity.
2. Transport all tubing to the field in precut, **precleaned** sections.
3. TEFLON, POLYETHYLENE AND POLYPROPYLENE TUBING
- 3.1. New Tubing: Follow this procedure unless the manufacturer/supplier provides certification that the tubing is clean.
- 3.1.1. Teflon
- 3.1.1.1. Rinse outside of tubing with pesticide-grade solvent (see FC 1001, section 2).

- 3.1.1.2. Flush inside of tubing with pesticide-grade solvent.
- 3.1.1.3. Dry overnight in drying oven or equivalent (zero air, nitrogen, etc.).
- 3.1.2. Polyethylene and Polypropylene
 - 3.1.2.1. Clean the exterior and interior of the tubing by soaking in hot, sudsy water.
 - 3.1.2.2. Thoroughly rinse the exterior and interior of the tubing with tap water, followed by analyte-free water.

3.2. Reused Tubing

Use the following procedure for in-lab cleaning. **Field cleaning is not recommended:**

- 3.2.1. Clean the exterior of the tubing by soaking in hot, sudsy water (see FC 1001, section 1) in a stainless steel sink (or equivalent non-contaminating material). Use a brush to remove any particulates, if necessary.
- 3.2.2. Use a small bottle brush and clean the inside of the tubing ends where the barbs are to be inserted or cut 1-2 inches from the ends of the tubing after cleaning.
- 3.2.3. Rinse tubing exterior and ends liberally with tap water.
- 3.2.4. Rinse tubing surfaces and ends with the appropriate acid solution (see FC 1001, section 4), tap water, isopropanol (see FC 1001, section 2), and finally analyte-free water.
 - 3.2.4.1. Note: Eliminate the isopropanol rinse for polyethylene or polypropylene tubing.
- 3.2.5. Place tubing on fresh aluminum foil or clean polyethylene sheeting. Connect all of the precut lengths of tubing with Teflon inserts or barbs.
- 3.2.6. Cleaning configuration:
 - 3.2.6.1. Place cleaning reagents: [sudsy water (see FC 1001, section 1); acid (see FC 1001, section 4); isopropanol (see FC 1001, section 2)] in an appropriately cleaned container (2-liter glass jar is recommended).
 - 3.2.6.2. Place one end of the Teflon tubing into the cleaning solution.
 - 3.2.6.3. Attach the other end of the Teflon tubing set to the influent end of a pump.
 - 3.2.6.4. Recycle the effluent from the pump by connecting a length of Teflon tubing from the effluent to the glass jar with the cleaning reagents.
 - 3.2.6.5. Recycling as described above may be done for all reagents listed in FC 1160, section 3.2.6.1 above, **except** the final isopropanol rinse and the final analyte-free water rinse. Disconnect the tubing between the effluent end of the pump and the jar of cleaning reagents.
 - 3.2.6.6. Containerize isopropanol in a waste container for proper disposal.
 - 3.2.6.7. Analyte-free water may be discarded down the drain.
- 3.2.7. Using the above configuration described in FS 1160, section 3.2.6 above:
 - 3.2.7.1. Pump hot, sudsy water through the connected lengths. Allow the pump to run long enough to pump at least three complete tubing volumes through the tubing set.

3.2.7.2. Using the same procedure, successively pump tap water, the acid solution(s), tap water, isopropanol, and finally analyte-free water through the system.

3.2.7.3. Leave the Teflon inserts or barbs between the precut lengths and cap or connect the remaining ends.

3.2.8. After the interior has been cleaned as described in FC 1160, section 3.2.7 above, rinse the exterior of the tubing with analyte-free water.

3.2.9. Wrap the connected lengths in aluminum foil or untreated butcher paper and store in a clean, dry area until use.

4. Flexible Tubing used in Pump Heads of Automatic Samplers and other Peristaltic Pumps

Replace tubing after each sampling point if samples are collected through the tubing. Unless the pump is deployed to collect samples from the same location over a long period of time, remove and wash the tubing after each sampling event (see FC 1140, section 1).

- 4.1. Flush tubing with hot tap water then sudsy water (see FC 1001, section 1).
- 4.2. Rinse thoroughly with hot tap water.
- 4.3. Rinse thoroughly with analyte-free water.
- 4.4. If used to collect metals samples, flush the tubing with an appropriate acid solution (see FC 1001, section 4), followed by thorough rinsing with analyte-free water. If used to collect both metals and nitrogen components use hydrochloric acid (see FC 1001, section 4.1.1).
- 4.5. Install tubing in peristaltic pump or automatic sampler.
- 4.6. Cap both ends with aluminum foil or equivalent.

Note: Change tubing at specified frequencies as part of routine preventative maintenance.

5. STAINLESS STEEL TUBING

Clean the exterior and interior of stainless steel tubing as follows:

- 5.1. Using sudsy water (see FC 1001, section 1), scrub the interior and exterior surfaces.
- 5.2. Rinse with hot tap water.
- 5.3. Rinse with analyte-free water.
- 5.4. If volatile or extractable organics are to be sampled, rinse all surfaces with isopropanol (see FC 1001, section 2). Use enough solvent to wet all surfaces with free flowing solvent.
- 5.5. Allow to air dry or thoroughly rinse with analyte-free water.

6. GLASS TUBING

- 6.1. Use new glass tubing.
- 6.2. If volatile or extractable organics are to be sampled, rinse with isopropanol (see FC 1001, section 2).
- 6.3. Air dry for at least 24 hours.
- 6.4. Wrap in aluminum foil or untreated butcher paper to prevent contamination during storage.

6.5. Discard tubing after use.

7. MISCELLANEOUS NON-INERT TUBING TYPES (TYGON, RUBBER, PVC, ETC.)

7.1. New Tubing

- 7.1.1. As a general rule, new tubing may be used without preliminary cleaning.
- 7.1.2. Protect new tubing from potential environmental contamination by wrapping in aluminum foil and sealing in untreated plastic bags or keep in the original sealed packaging until use.
- 7.1.3. If new tubing is exposed to potential contamination, rinse the exterior and interior tubing surfaces with hot tap water followed by a thorough rinse with analyte-free water.
- 7.1.4. If new tubing is to be used to collect samples, thoroughly rinse the tubing with sample water (i.e., pump sample water through the tubing) before collecting samples.

7.2. Reused Tubing

- 7.2.1. Flush tubing with sudsy solution of hot tap water and laboratory detergent (see FC 1001, section 1).
- 7.2.2. Rinse exterior and interior thoroughly with hot tap water.
- 7.2.3. Rinse exterior and interior thoroughly with analyte-free water.
- 7.2.4. If used to collect only metals samples, flush the tubing with nitric acid (see FC 1001, section 4.1), followed by a thorough rinse with analyte-free water.
- 7.2.5. If used to collect metals and nitrogen-containing compounds, see FC 1001, section 4.3.
- 7.2.6. Cap ends in aluminum foil and store in clean, untreated plastic bags to prevent contamination during storage and transport.

FC 1170. PUMPS

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1. ~~SUBMERSIBLE PUMPS~~

1.1. ~~Pumps used for Purging and Sampling Metals and/or Volatile and Extractable Organics~~

- 1.1.1. Construction of pump body and internal mechanisms (bladders, impellers, etc.), including seals and connections, must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.
- 1.1.2. Tubing material must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.
- 1.1.3. Clean pump exterior following FC 1132. Note: omit the solvent rinse if the pump body is constructed of plastic (e.g., ABS, PVC, etc.).
- 1.1.4. Clean the pump internal cavity and mechanism as follows:
 - 1.1.4.1. If used only for purging, thoroughly flush the pump with water before purging the next well.
 - 1.1.4.2. When used for purging and sampling, completely disassemble the pump (if practical) and decontaminate between each well.
 - 1.1.4.3. When used for purging and sampling and the pump cannot be (practicably) disassembled, then clean the internal cavity/mechanism by pumping

several gallons of sudsy water (see FC 1001, section 1), followed by several gallons of tap water, and finally, several gallons of analyte-free water.

1.1.4.4. If multiple sampling points are located in an area that is not accessible by a vehicle, and it is difficult to reach a vehicle for cleaning or to transport all cleaning materials to the staging area, at a minimum thoroughly rinse the pump with water. TD
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1.1.5. Refer to FC 1160, section 3 to clean Teflon tubing.

1.1.6. Refer to FC 1160, section 5 for stainless steel tubing.

1.1.7. Clean other types of tubing according to FC 1160, sections 6 and 7.

1.2. Pumps used for Purging and Sampling all Analytes except Metals, Volatile and Extractable Organics

1.2.1. Pump construction: no restrictions.

1.2.2. Pump tubing material: no restrictions.

1.2.3. Scrub the exterior of the pump with appropriate metal-free, phosphate-free or ammonia-free detergent solution.

1.2.4. Rinse the exterior with tap water and analyte-free water.

1.2.5. Rinse the interior of the pump and tubing by pumping tap or analyte-free water through the system using a clean bucket or drum.

2. ABOVE-GROUND PUMPS USED FOR PURGING AND SAMPLING

2.1. Pumps used only for Purging

2.1.1. The exterior of the pump must be free of oil and grease.

2.1.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.1.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

2.2. Pumps used for Sampling

2.2.1. Clean the exterior of the pump with a detergent solution followed by a tap water rinse. Use clean cloths or unbleached paper towels that have been moistened with the appropriate solution to wipe down the pump.

2.2.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.2.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

FC 1180. ANALYTE-FREE WATER CONTAINERS

This section pertains to containers that are purchased to transport, store and dispense analyte-free water. It does not apply to water that has been purchased in containers. See FC 1002, section 3 for appropriate construction materials.

1. NEW CONTAINERS

1.1. Wash containers and caps according to FC 1131, omitting the solvent rinse if plastic (polyethylene or polypropylene) containers are being cleaned.

1.2. Cap with Teflon film or the bottle cap. The bottle cap must be composed of the same material as the container and cannot be lined.

2. REUSED CONTAINERS

2.1. Immediately after emptying, cap with aluminum foil, Teflon film or the container cap.

2.2. Wash the exterior of the container with lab-grade detergent solution (see FC 1001, section 1) and rinse with analyte-free water.

2.3. Rinse the interior thoroughly with analyte-free water.

2.4. Invert and allow to drain and dry.

FC 1190. ICE CHESTS AND SHIPPING CONTAINERS

1. Wash the exterior and interior of all ice chests with laboratory detergent (see FC 1001, section 1) after each use.

2. Rinse with tap water and air dry before storing.

3. If the ice chest becomes severely contaminated with concentrated waste or other toxic or hazardous materials clean as thoroughly as possible, render unusable, and properly dispose.

FC 1200. Field Instruments and Drilling Equipment

FC 1210. FIELD INSTRUMENTS (TAPES, METERS, ETC.)

Follow manufacturer's recommendations for cleaning instruments. At a minimum:

1. Wipe down equipment body, probes, and cables with lab-grade detergent solution (see FC 1001, section 1). Check manufacturer's instructions for recommendations and/or restrictions on cleaning.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte-free water.

4. Store equipment according to the manufacturer's recommendation or wrap equipment in aluminum foil, untreated butcher paper or untreated plastic bags to eliminate potential environmental contamination.

FC 1220. SOIL BORING EQUIPMENT

This section pertains only to equipment that is not used to collect samples. Clean split spoons, bucket augers and other sampling devices according to FC 1131.

1. Remove oil, grease, and hydraulic fluid from the exterior of the engine and power head, auger stems, bits and other associated equipment with a power washer or steam jenny or wash by hand with a brush and sudsy waster (no degreasers).

2. Rinse thoroughly with tap water.

FC 1230. WELL CASING CLEANING

These are recommended procedures for cleaning well casing and riser pipes. Use procedures specified by a FDEP contract, order, permit, or rule, if different or more stringent than the procedures outlined below.

1. FDEP recommends only using casing that is designed for subsurface environmental groundwater monitoring.
2. Casing that has been contaminated with grease, hydraulic fluid, petroleum fuel, etc. may require additional cleaning or deemed unusable.
3. All casings and riser pipes should be cleaned before installation, unless the casing is received wrapped and ready for installation:
 - 3.1. Steam clean all casings and riser pipes except PVC. Steam cleaning criteria shall meet the following: water pressure - 2500 psi; water temperature - 200°F.
 - 3.2. Rinse thoroughly with tap (potable) water. This tap water must be free of the analytes of interest.

FC 1300. Sample Containers

FC 1310. OBTAINING CLEAN CONTAINERS

1. Obtain clean sample containers in one of three ways:
 - 1.1. From commercial vendors as precleaned containers. The cleaning grades must meet EPA analyte specific requirements. Keep all records for these containers (lot numbers, certification statements, date of receipt, etc.) and document the container's intended uses;
 - 1.2. From internal groups within the organization that are responsible for cleaning and maintaining containers according to the procedures outlined in FC 1320; or
 - 1.3. From a subcontracted laboratory that is accredited under the National Environmental Laboratory Accreditation Program (NELAP).
 - 1.3.1. The contractor must verify that the laboratory follows the container cleaning procedures outlined in FC 1320.
 - 1.3.2. If the laboratory cleaning procedures are different, the contractor must require that the laboratory use the following cleaning procedures or provide documentation and historical records to show that their in-house procedure produces containers that are free from the analytes of interest.

FC 1320. CONTAINER CLEANING PROCEDURES

1. Refer to Table FC 1000-2. Follow the cleaning steps in the order specified in the chart.
2. Cleaning procedures that are different from those outlined in FC 1320 may be used as long as blanks collected in the containers are free from the analytes of interest and any analytical interferences and the cleaning procedures are supported by historical and continuing documentation.
3. Inspect all containers before cleaning.
 - 3.1. **Do not recycle or reuse containers if:**
 - 3.1.1. Containers were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;
 - 3.1.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or

3.1.3. Containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers shall be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.

3.1.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest before use. If found to be contaminated (i.e., analytes of interest are found at MDL levels or higher), discard the containers.

FC 1400. Documentation

Document cleaning procedures described below for the indicated activities. See FD 1000 for additional information about required records and retention of documents.

FC 1410. FIELD EQUIPMENT

1. IN-FIELD CLEANING

1.1. Initially identify the procedures that are used to clean equipment in the field by SOP numbers and dates of usage.

1.2. Record the date and time that equipment was cleaned.

2. IN-HOUSE CLEANING

2.1. Retain any cleaning certificates, whether from a laboratory or commercial vendor.

2.2. Identify the procedure(s) that are used to clean equipment by the SOP number and dates of usage.

2.3. Record the date that the equipment was cleaned.

FC 1420. SAMPLE CONTAINERS

1. Organizations that order precleaned containers must retain the packing slips, and lot numbers of each shipment, any certification statements provided by the vendor and the vendor cleaning procedures.

2. Organizations that clean containers must maintain permanent records of the following:

2.1. Procedure(s) used to clean containers by SOP number and dates of usage.

2.2. If containers are certified clean by the laboratory the laboratory must record:

- Type of container;
- Date cleaned;
- SOP used;
- Person responsible for cleaning;
- Lot number (date of cleaning may be used) of the batch of containers that were cleaned using the same reagent lots and the same procedure;
- The results of quality control tests that were run on lot numbers; and
- Any additional cleaning or problems that were encountered with a specific lot.

FC 1430. REAGENTS AND OTHER CLEANING SUPPLIES

Maintain a record of the lot number with the inclusive dates of use for all acids, solvents, and other cleaning supplies.

Appendix FC 1000
Tables, Figures and Forms

Table FC 1000-1 Procedures for Decontamination at the Base of Operations or On-site

Table FC 1000-2 Container Cleaning Procedures

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
Teflon or Glass	All	FC 1131	Follow as written	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Extractable & Volatile Organics Petroleum Hydrocarbons		May omit acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit acid rinse
	Metals ¹ Radionuclides For ultra trace metals, refer to FS 8200		May omit solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit solvent rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Metallic (stainless steel, brass, etc.)	All Extractable & Volatile Organics Petroleum Hydrocarbons	FC 1131	Omit the acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse
	Metals Radionuclides		Omit the acid rinse May omit the solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse May omit the solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		Omit solvent rinse May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Plastic (Polyethylene, polypropylene, PVC, silicone, acrylic)	Volatile and Extractable Organics;	FC 1132	Follow as written.	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

ⁱ Do not use glass if collecting samples for boron or silica.

Table FC 1000-2
Container Cleaning Procedures

ANALYSIS / ANALYTE GROUP	CLEANING STEPS See Description Below
Extractable Organics	1, 2, 4, 6 (not required if Luminox (or equivalent is used), (5 and 7 optional), 11
Volatile Organics	1, 2, 4, (6 optional, methanol only), 7
Metals	1, 2, 3, 4, 8, 11 ** **Procedures to clean containers for ultra-trace metals are found in FS 8200
Inorganic Nonmetallics, Radionuclides, Physical and Aggregate Properties, Aggregate Inorganics, and Volatile Inorganics	1, 2, 3*, 4, 8, 11 * For nutrients, replace nitric acid with hydrochloric acid, or use a hydrochloric acid rinse after the nitric acid rinse. See FC 1001, section 4
Petroleum Hydrocarbons, and Oil and Grease	1, 2, 3, 4, (5, 6, 7 optional), 11
Microbiological (all)	1, 2, 4, 8, 9, 11
Toxicity Tests (Includes Bioassays)	1, 2, 10, 2, 4, 6.1, (10 optional), 11

NOTE: Steps 1 and 2 may be omitted when cleaning new, uncertified containers.

1. Wash with hot tap water and a brush using a suitable laboratory-grade detergent:
 - 1.1. Volatile and Extractable Organics, Petroleum Hydrocarbon, Oil and Grease: Luminox, Liqui-Nox, Alconox or equivalent;
 - 1.2. Inorganic nonmetallics: Liqui-Nox or equivalent;
 - 1.3. Metals: Liqui-Nox, Acationox, Micro or equivalents;
 - 1.4. Microbiologicals (all): Must pass an inhibitory residue test.
2. Rinse thoroughly with hot tap water.
3. Rinse with 10% nitric acid solution.
4. Rinse thoroughly with analyte-free water (deionized or better).
5. Rinse thoroughly with pesticide-grade methylene chloride.
6. Rinse thoroughly with pesticide-grade isopropanol, acetone or methanol.
 - 6.1. For bioassays, use only acetone, and only when containers are glass.
7. Oven dry at 103°C to 125°C for at least 1 hour.

Table FC 1000-2
Container Cleaning Procedures

- 7.1. VOC vials and containers must remain in the oven in a contaminant-free environment until needed. They should be capped in a contaminant-free environment just prior to dispatch to the field.
8. Invert and air-dry in a contaminant-free environment.
9. Sterilize containers:
 - 9.1. Plastic: 60 min at 170°C, loosen caps to prevent distortion.
 - 9.2. Glass: 15 min at 121°C.
10. Rinse with 10% hydrochloric acid followed by a sodium bicarbonate solution.
11. Cap tightly and store in a contaminant-free environment until use. Do not use glass if collecting samples for boron or silica.

FD 1000. DOCUMENTATION PROCEDURES

1. INTRODUCTION:

1.1. For the creation of clear, accurate and methodical records to document all field activities affecting sample data, implement the following standard operating procedures for sample collection, sample handling and field-testing activities.

2. SCOPE AND APPLICABILITY

2.1. This SOP provides a detailed listing of the information required for documentation of all sampling procedures and field testing.

2.2. Refer to the associated sampling or field testing SOP for any requirements for the chronological or sequential documentation of data.

3. QUALITY ASSURANCE

3.1. Implement review procedures to monitor and verify accurate manual and automated data entry and recordkeeping for all documentation tasks outlined in this SOP.

FD 1100. Universal Documentation Requirements

Incorporate efficient archival design and concise documentation schemes for all record systems. Ensure that the history of a sample is clearly evident in the retained records and documentation and can be independently reconstructed.

1. CRITERIA FOR ALL DOCUMENTS

1.1. Keep all applicable documentation available for inspection. Keep all original data and records as well as reduced or manipulated forms of the original data or records.

1.1.1. Authorized representatives of DEP have the legal right to inspect and request copies of any records using paper, electronic media, or other media during any DEP audit of physical facilities or on-site sampling events, and for any data validations conducted for applicable project data submitted to DEP.

1.2. Record enough information so that clarifications, interpretations, or explanations of the data are not required from the originator of the documentation.

1.3. Clearly indicate the nature and intent of all documentation and all record entries.

1.4. Link citations to SOPs and other documents by the complete name, reference or publication number, revision number, and revision date for the cited document, when applicable. Also assign this information to internally generated SOPs.

1.5. Retain copies of all revisions of all cited documents as part of the documentation archives.

2. PROCEDURES

2.1. Sign, initial or encode all documentation entries made to paper, electronic or other records with a link indicating the name and responsibility of the author making the data entry, clearly indicating the reason for the signature, initials or code (e.g., "sampled by"; "released by"; "prepared by"; "reviewed by").

2.2. In order to abbreviate record entries, make references to procedures written in internal SOPs or methodology and procedures promulgated by external sources.

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2.2.1. Document the intent to use SOPs other than the DEP SOPs, or to use allowable modifications to the DEP SOPs by recording the effective date of use for all such SOPs or modifications.

2.2.1.1. Retain any correspondence with DEP regarding approval to use alternative procedures for any projects.

2.2.2. Authorize all internal SOPs with the signatures of the quality assurance officer(s) and manager(s) responsible for implementation of the SOPs. Record the dates of signature.

2.3. Employ straightforward archiving of records to facilitate documentation tracking and retrieval of all current and archived records for purposes of inspection, verification, and historical reconstruction of all procedures and measurement data.

2.4. Keep copies or originals of all documentation, including documentation sent to or received from external parties.

2.5. Use waterproof ink for all paper documentation.

2.6. Do not erase or obliterate entry errors on paper records. Make corrections by marking a line through the error so that it is still legible. Initial or sign the marked error and its correction.

2.7. Maintain electronic audit trails for all edited electronic records, if possible. Utilize software that allows tracking of users and data edits, if available. Software that prompts the user to double-check edits before execution is also preferred. See FD 1200.

2.8. Clearly link all documentation associated with a sample or measurement. Make cross-references to specific documentation when necessary.

2.9. Link final reports, data summaries, or other condensed versions of data to the original sample data, including those prepared by external parties.

3. RETENTION REQUIREMENTS

3.1. Per the DEP QA Rule, 62-160.220 & .340, F.A.C., keep all documentation archives for a minimum of 5 years after the date of project completion or permit cycle unless otherwise specified in a Department contract, order, permit, or Title 62 rules.

FD 1200. Electronic Documentation

Handle electronic (digital) data as with any data according to applicable provisions of FD 1100.

1. RETENTION OF AUTOMATIC DATA RECORDING PRODUCTS

1.1. For data not directly read from the instrument display and manually recorded, retain all products or outputs from automatic data recording devices, such as strip chart recorders, integrators, data loggers, field measurement devices, computers, etc. Store records in electronic, magnetic, optical, or paper form, as necessary.

1.1.1. Retain all original, raw output data. Ensure archiving of these data prior to subsequent reduction or other manipulation of the data.

1.2. Identify output records as to purpose, analysis date and time, field sample identification number, etc. Maintain clear linkage with the associated sample, other data source or measured medium and specific instrument used to make the measurement.

2. ELECTRONIC DATA SECURITY

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- 2.1. Control levels of access to electronic data systems as required to maintain system security and to prevent unauthorized editing of data.
 - 2.2. Do not alter raw instrumentation data or original manual data records in any fashion without retention of the original raw data.
 - 2.3. Maintain secure computer networks and appropriate virus protection as warranted for each system design.
3. ELECTRONIC DATA STORAGE AND DOCUMENTATION
- 3.1. Store all electronic, magnetic, and optical media for easy retrieval of records.
 - 3.1.1. Ensure that all records can be printed to paper if needed for audit or verification purposes.
 - 3.1.2. If it is anticipated that the documentation archive will become unreadable due to obsolescence of a particular storage technology, retain a paper archive of the data or transfer to other suitable media.
 - 3.2. For easy retrieval of records, link all stored data to the associated sample data or other data source.
 - 3.3. Back up all data at a copy rate commensurate with the level of vulnerability of the data. Consider replicating all original data as soon as possible after origination.
4. SOFTWARE VERIFICATION
- 4.1. Ensure that any software used to perform automatic calculations conforms to required formulas or protocols.
 - 4.2. Document all software problems and their resolution in detail, where these problems have irretrievably affected data records or linkage. Record the calendar date, time, responsible personnel, and relevant technical details of all affected data and software files. Note all software changes, updates, installations, etc. per the above concerns. File and link all associated service records supplied by vendors or other service personnel.
5. PROTECTION OF EQUIPMENT AND STORAGE MEDIA
- 5.1. Place stationary computers, instrumentation, and peripheral devices in locations of controlled temperature and humidity and away from areas where the potential for fluid leaks, fire, falling objects, or other hazards may exist. In the field, protect portable equipment from weather, excess heat or freezing, storage in closed vehicles, spillage from reagents and samples, etc.
 - 5.2. Protect storage media from deteriorating conditions such as temperature, humidity, magnetic fields, or other environmental hazards as above.
6. ELECTRONIC SIGNATURES – Documents signed with electronic signatures must be consistent with the requirements of 62-160.405, F.A.C.:
- 6.1. the integrity of the electronic signature can be assured;
 - 6.2. the signature is unique to the individual;
 - 6.3. the organization using electronic signatures has written policies for the generation and use of electronic signatures; and
 - 6.4. the organization using electronic signatures has written procedures for ensuring the security, confidentiality, integrity and auditability of each signature.

FD 1300. Documentation Using Other Media

1. UNIVERSAL REQUIREMENTS

1.1. Handle documentation prepared using other media according to FD 1100.

2. PROTECTION OF STORED MEDIA

2.1. Store media such as photographs, photographic negatives, microfilm, videotape, etc. under conditions generally prescribed for these media by manufacturers and conducive to long-term storage and protection from deterioration. See also FD 1200, section 5, above.

FD 2000. DOCUMENTATION OF CLEANED EQUIPMENT, SAMPLE CONTAINERS, REAGENTS AND SUPPLIES

When providing sample containers, preservation reagents, analyte-free water or sampling equipment, document certain aspects of these preparations.

1. EQUIPMENT CLEANING DOCUMENTATION

1.1. Document all cleaning procedures by stepwise description in an internal SOP if cleaning procedures in the DEP SOP have been modified for use. Alternatively, cite the DEP SOP procedures in the cleaning record for the applicable equipment.

1.2. Record the date of cleaning.

1.2.1. If items are cleaned in the field during sampling activities for a site, document the date and time when the affected equipment was cleaned. Link this information with the site and the cleaning location at the site.

1.3. Retain or make accessible any certificates of cleanliness issued by vendors supplying cleaned equipment or sample containers.

1.3.1. Retain from the vendor or document for internal cleaning the following information for sample containers, as applicable:

- Packing slip and cleanliness certificates from vendors
- Container types and intended uses
- Lot numbers or other designations for groups of containers cleaned together using the same reagents and procedures
- Dates of cleaning
- Cleaning procedures or reference to internal cleaning SOPs or DEP SOPs
- Cleaning personnel names
- Results of quality control analyses associated with container lots
- Comments about problems or other information associated with container lots

2. SAMPLING KIT DOCUMENTATION

If supplied to a party other than internal staff, transmit to the recipient the following information pertaining to sampling equipment or other implements, sample containers, reagent containers, analyte-free water containers, reagents or analyte-free water supplied to the recipient.

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- Quantity, description and material composition of all containers, container caps or closures or liners for caps or closures
 - Intended application for each sample container type indicated by approved analytical method or analyte group(s)
 - Type, lot number, amount and concentration of preservative added to clean sample containers and/or shipped as additional preservative
 - Intended use for any additional preservatives or reagents provided
 - Description of any analyte-free water (i.e., deionized, organic-free, etc.)
 - Date of analyte-free water containerization
 - Date of sampling kit preparation
 - Description and material composition of all reagent transfer implements (e.g., pipets) shipped in the sampling kit and the analyte groups for which the implements have been cleaned or supplied
 - Quantity, description and material composition of all sampling equipment and pump tubing (including equipment supplied for filtration) and the analyte groups for which the equipment has been cleaned or supplied
 - Tare weight of VOC vials, as applicable (this item is necessary when EPA 5035 VOC sample vials are provided for soil samples)
3. DOCUMENTATION FOR REAGENTS AND OTHER CHEMICALS
- 3.1. Keep a record of the lot numbers and inclusive dates of use for all reagents, detergents, solvents and other chemicals used for cleaning and sample preservation.
- 3.1.1. See FD 4000 below for documentation requirements for reagents used for field testing.

FD 3000. DOCUMENTATION OF EQUIPMENT MAINTENANCE

1. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures, corrective actions performed during calibrations or verifications, and solution or parts replacement for instrument probes.
 - 1.1. Include the calendar date for the procedures performed.
 - 1.2. Record names of personnel performing the maintenance or repair tasks.
 - 1.2.1. Describe any malfunctions necessitating repair or service.
2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number, or other unique identification.
3. Retain vendor service records for all affected instruments.
4. Record the following for rented equipment:

- Rental date(s)
 - Equipment type and model or inventory number or other description
5. Retain the manufacturer's operating and maintenance instructions.

FD 4000. DOCUMENTATION FOR CALIBRATION OF FIELD-TESTING INSTRUMENTS AND FIELD ANALYSES

Document acceptable instrument or measuring system calibration for each field test or analysis of a sample or other measurement medium.

FD 4100. General Documentation for all Field Testing

1. STANDARD AND REAGENT DOCUMENTATION: Document information about standards and reagents used for calibrations, verifications, and sample measurements.
 - 1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.
 - 1.1.1. Document acceptable verification of any standard used after its expiration date.
 - 1.2. Record the concentration or other value for the standard in the appropriate measurement units.
 - 1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.
 - 1.2.2. Retain vendor assay specifications for standards as part of the calibration record.
 - 1.2.2.1. Record the grade of standard or reagent used.
 - 1.3. When formulated in-house, document all calculations used to formulate calibration standards.
 - 1.3.1. Record the date of preparation for all in-house formulations.
 - 1.4. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).
2. FIELD INSTRUMENT CALIBRATION DOCUMENTATION: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.
 - 2.1. Retain vendor certifications of all factory-calibrated instrumentation.
 - 2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.
 - 2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
 - 2.3. Record the time and date of all initial calibrations and all calibration verifications.
 - 2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
 - 2.5. Record the name of the analyst(s) performing the calibration or verification.

2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Link to information recorded according to section 1 above

2.7. Retain manufacturers' instrument specifications.

2.8. Document whether successful initial calibration occurred.

2.9. Document whether each calibration verification passed or failed.

2.10. Document, according to records requirements of FD 3000, any corrective actions taken to modify instrument performance.

2.10.1. Document date and time of any corrective actions.

2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- "J" data qualifier code for estimated measurement or test sample value
- Reporting units for the measurement
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit used for the test (see 2.2 above)

FD 5000. DOCUMENTATION OF SAMPLE COLLECTION, PRESERVATION AND TRANSPORT

Follow these procedures for all samples. See FD 5100 - FD 5427 below for additional documentation for specific sampling activities. See example Forms in FD 9000 below for example formats for documenting specific sampling and testing procedures.

1. SAMPLE IDENTIFICATION REQUIREMENTS

1.1. Ensure that labels are waterproof and will not disintegrate or detach from the sample container when wet, especially under conditions of extended submersion in ice water typically accumulating in ice chests or other transport containers.

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1.2. Label or tag each sample container with a unique field identification code that adequately distinguishes each sample according to the following criteria. The code must adequately link the sample container with all of the information about the sample contained in the permanent field record.

1.2.1. Link the unique field identification code to the sample source or sampling point identification, the date of sample collection, the time of sample collection (for maximum holding times equal to or less than 48 hours), the analytes of interest and the preservation technique.

1.2.2. Label or tag each sample container for the following types of samples with a unique field identification code:

- Quality control samples such as duplicate samples, other replicate samples or split samples collected for the same analyte or group of analytes
- Field samples or quality control samples collected using a different sample collection technique for the same analyte or group of analytes (for example, if both a bailer and a pump are used to collect samples for metals analysis, label the bailer sample to distinguish it from the pump sample)

1.2.3. The color, size, shape, or material composition of sample containers and caps cannot substitute for the information required in 1.2.1. – 1.2.2. Above.

1.2.4. The unique field identification code and any other information included on the container label or tag must allow the analyzing laboratory to independently determine the sample collection date, the sample collection time (for maximum holding times \leq 48 hours), the sample preservation and the analytical tests to be performed on each container or group of containers.

1.3. Attach the label or tag so that it does not contact any portion of the sample that is removed or poured from the container.

1.4. Record the unique field identification code on all other documentation associated with the specific sample container or group of containers.

2. GENERAL REQUIREMENTS FOR SAMPLING DOCUMENTATION: Record the following information for all sampling:

2.1. Names of all sampling team personnel on site during sampling

2.2. Date and time of sample collection (indicate hours and minutes)

2.2.1. Use 24-hour clock time or indicate A.M. and P.M.

2.2.2. Note the exact time of collection for individual sample containers for time-sensitive analyses with a maximum holding time of 48 hours or less.

2.3. Ambient field conditions, to include, but not limited to information such as weather, tides, etc.

2.4. Comments about samples or conditions associated with the sample source (e.g., turbidity, sulfide odor, insufficient amount of sample collected)

2.5. Specific description of sample location, including site name and address

2.5.1. Describe the specific sampling point (e.g., monitoring well identification number, outfall number, station number, etc.).

2.5.2. Determine latitude and longitude of sampling source location (if required).

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- 2.5.3. Locate sampling points on scaled maps or drawings where applicable.
 - 2.6. Record the unique field identification code for each sample container and parameters to be analyzed, per section 1 above. The code must adequately link the sample container or group of containers with all of the information about the sample contained in the permanent field record.
 - 2.7. Number of containers collected for each unique field identification code
 - 2.8. Matrix sampled
 - 2.9. Type of field sample collected, such as grab, composite or other applicable designation.
 - 2.10. Field-testing measurement data:
 - 2.10.1. See FD 4000 above for specific details.
 - 2.11. Calibration records for field-testing equipment
 - 2.11.1. See FD 4000 above for specific details.
 - 2.12. Preservation for each container
 - 2.12.1. Indicate whether samples are chemically preserved on-site by the sampling team or, alternatively, were collected in prepreserved (predosed) containers.
 - 2.12.2. Indication of any tests performed in the field to determine the presence of analytical interferences in the sample.
 - 2.12.3. Indication of any treatments of samples performed in the field to eliminate or minimize analytical interferences in the sample.
 - 2.12.4. See FD 5100, section 1.
 - 2.13. Purging and sampling equipment used, including the material composition of the equipment and any expendable items such as tubing.
 - 2.14. Types, number, collection location and collection sequence of quality control samples
 - 2.14.1. Include a list of equipment that was rinsed to collect any equipment blanks.
 - 2.15. Use of fuel powered vehicles and equipment
 - 2.16. Number of subsamples and amount of each subsample in any composite samples
 - 2.16.1. Include sufficient location information for the composite subsamples per 2.4 above.
 - 2.17. Depth of all samples or subsamples
 - 2.18. Signature(s) or initials of sampler(s)
3. SAMPLE TRANSMITTAL RECORDS: Transmit the following information to the analytical laboratory or other receiving party. Link transmittal records with a given project and retain all transmittal records.
- Site name and address – Note: Client code is acceptable if samples are considered sensitive information and if the field records clearly trace the code to a specified site and address.
 - Date and time of sample collection

- Name of sampler responsible for sample transmittal
- Unique field identification codes for each sample container
- Total number of samples
- Required analyses
- Preservation protocol
- Comments about sample or sample conditions
- Identification of common carrier (if used)

4. SAMPLE TRANSPORT

4.1. If shipping transmittal forms in the transport containers with the samples, place the forms in a waterproof enclosure and seal.

4.2. For common carrier shipping, seal transport containers securely with strapping tape or other means to prevent lids from accidentally opening.

4.2.1. Keep all shipping bills from common carriers with archived transmittal records.

5. ANCILLARY FIELD RECORDS: Link any miscellaneous or ancillary records (photographs, videotapes, maps, etc.) to specific sampling events such that these records are easily traceable in the data archives associated with the project, sampling date and sample source(s).

FD 5100. Documentation Specific To Aqueous Chemistry Sampling

1. SAMPLE PRESERVATION: Document preservation of all samples according to the following instructions.

1.1. List the chemical preservatives added to the sample.

1.2. Record the results of pH verification performed in the field, including the pH value of the sample (if applicable). Note any observations about changes in the sample as a result of adding preservative to the sample or mixing the sample with the preservative.

1.3. Record the amount of preservative added to samples and the amount of any additional preservative added. The amount dosed into sample containers supplied with premeasured preservatives must also be recorded.

1.3.1. For documentation of procedures for preservation for routine samples, cite DEP SOPs or internal SOPs for this information.

1.3.2. Record instances of deviation from preservation protocols found in SOPs when non-routine or problematic samples are collected.

1.4. Record the use of ice or other cooling method, when applicable.

2. GROUNDWATER SAMPLING

2.1. Record or establish a documentation link to the following information for all samples. See section 3 below for in-place plumbing:

- Well casing composition and diameter of well casing
- A description of the process and the data used to design the well

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- The equipment and procedure used to install the well
 - The well development procedure
 - Pertinent lithologic or hydrogeologic information
 - Ambient conditions at the wellhead or sampling point that are potential sources of unrepresentative sample contamination
 - Water table depth and well depth
 - Calculations used to determine purge volume
 - Total amount of water purged
 - Date well was purged
 - Purging equipment used
 - Sampling equipment used
 - Well diameter
 - Total depth of well
 - Depth to groundwater
 - Volume of water in the well
 - Purging method
 - Placement depth of tubing or pump intake
 - Depth and length of screened interval
 - Times for beginning and ending of purging
 - Total volume purged
 - Times of stabilization parameter measurements
 - Purging rate, including any changes in rate
 - Temperature measurements
 - pH measurements
 - Specific conductance measurements
 - Dissolved oxygen measurements
 - Turbidity measurements
 - Site or monitoring well conditions impacting observed dissolved oxygen and turbidity measurements
 - Color of groundwater
 - Odor of groundwater
- 2.2. Record the following for Water Level and Purge Volume Determination (FS 2211):
- Depth to groundwater
 - Total depth of well

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- Length of water column
- Well diameter
- Volume of water in the well
- Volume of pump
- Tubing diameter
- Length of tubing
- Volume of flow cell
- Volume in the pumping system

2.3. Record the following for Well Purging (FS 2212)

- Calculations for pumping rates, including any changes in rates
- Flow meter readings
- Volume of water purged
- Placement depth of tubing or pump intake
- Depth and length of screened interval
- Time needed to purge one (1) well volume or purging equipment volume
- Well volumes or purging equipment volumes purged
- Temperature measurements
- pH measurements
- Specific conductance measurements
- Dissolved oxygen measurements
- Turbidity measurements
- Purging rate, including any changes in rate
- Drawdown in the well

3. IN-PLACE PLUMBING SOURCES INCLUDING DRINKING WATER SYSTEMS

3.1. Record the following for all samples:

- Plumbing and tap material construction (if known)
- Flow rate at which well was purged
- Amount of time well was allowed to purge
- Flow rate at time of sample collection
- Public water system identification number (if applicable)
- Name and address of water supply system and an emergency phone number for notification of sample results (if applicable)

4. SURFACE WATER SAMPLING

- Sample collection depth

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- Beginning and ending times (24 hr) for timed composite sampling
- Type of composite (e.g., flow-proportioned, continuous, etc.)

5. WASTEWATER SAMPLING

- Beginning and ending times (24 hr) for timed composite sampling
- Type of composite (e.g. flow-proportioned, continuous, etc.)

FD 5120. RECORDS FOR NON-AQUEOUS ENVIRONMENTAL SAMPLES

Document the following information for all samples when using the indicated procedures.

FD 5130. DOCUMENTATION SPECIFIC TO SOIL SAMPLING (FS 3000)

1. GENERAL SOIL SAMPLING

- Sample collection depth
- Areal location of sample
- Sample collection device

2. Sampling for Volatile Organic Compounds (VOC) per EPA Method 5035

- Tare weight of VOC sample vial (if applicable)
- Weight of sample (if applicable)

FD 5140. DOCUMENTATION SPECIFIC TO SEDIMENT SAMPLING (FS 4000)

1. General Sediment Sampling

- Sample collection depth
- Areal location of sample
- Sample collection device

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2. Sampling for Volatile Organic Compounds (VOC) per EPA Method 5035

- Tare weight of VOC sample vial (if applicable)
- Weight of sample (if applicable)

FD 5200. Documentation Specific to Waste Sampling (FS 5000)

1. DRUM SAMPLING

1.1. Record the following information for each drum:

- Type of drum and description of contents
- Drum number, if applicable
- Terrain and drainage condition
- Shape, size and dimensions of drum
- Label wording or other markings

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- Dimensional extent of leaks or spills associated with the drum
- Drum location (or location map)
- 1.2. Record the following information for the drum sample(s):
 - Description of phases, colors, crystals, powders, sludges, etc.
 - Stratified layers sampled, including aliquot amounts for composites, if applicable
- 1.3. Record the following for field testing results on opened drums and drum samples:
 - Background readings for OVA meters
 - Sample readings for OVA meters
 - Type of OVA probe
 - Radiation background reading and sample radiation reading
 - Type of radiation monitor used
 - Oxygen and LEL readings from container opening
 - Water reactivity results
 - Specific gravity
 - PCB test results
 - Water solubility results
 - pH of aqueous wastes
 - Results of chemical test strips
 - Ignitability results
 - Results of other chemical hazard test kits
 - Miscellaneous comments for any tests
- 2. Documentation for Tanks
 - 2.1. Record the following information for the tank:
 - Type of tank, tank design and material of construction of tank
 - Description of tank contents and markings
 - Tank number or other designation, if applicable
 - Terrain and drainage condition
 - Shape, size and dimensions of tank
 - Label or placard wording or other markings
 - Dimensional extent of leaks or spills associated with the tank
 - Tank location (or location map)
 - 2.2. Record the following information for the tank sample(s):
 - Description of phases, colors, crystals, powders, sludges, etc.

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- Stratified layers sampled, including aliquot amounts for composites, if applicable
- 2.3. Record the following for field testing results on opened tanks and tank samples:

- Background readings for OVA meters
- Sample readings for OVA meters
- Type of OVA probe
- Radiation background reading and sample radiation reading
- Type of radiation monitor used
- Oxygen and LEL level from container opening
- Water reactivity results
- Specific gravity
- PCB test results
- Water solubility results
- pH of aqueous wastes
- Results of chemical test strips
- Ignitability results
- Results of other chemical hazard test kits
- Miscellaneous comments for any tests

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3. DOCUMENTATION FOR WASTE LEACHATE AND WASTE SUMP SAMPLES

3.1. Document information specific to leachate and sump sampling according to the documentation requirements for the respective DEP SOPs employed to collect samples (FS 2100, FS 2200, FS 4000, FS 5100 and FS 5200).

4. DOCUMENTATION FOR WASTE PILE SAMPLES

4.1. Document information specific to waste pile sampling according to associated regulatory requirements for the project.

5. DOCUMENTATION FOR WASTE IMPOUNDMENT AND WASTE LAGOON SAMPLES

5.1. Document information specific to impoundment and lagoon sampling according to the documentation requirements for the respective DEP SOPs employed to collect samples (FS 2100, FS 4000, FS 5100, and FS 5200).

FD 5300. Documentation for Biological Sampling

The following SOP sections list required documentation items for specific biological sampling procedures, as indicated.

FD 5310. DOCUMENTATION FOR BIOLOGICAL AQUATIC HABITAT CHARACTERIZATION

Minimum documentation required for biological habitat characterization and sampling is listed below according to requirements as specified in the indicated sampling and field-testing DEP SOPs.

~~FD 5311. Physical/Chemical Characterization for Biological Sampling (FT 3001)~~

1. Record the following information or use the Physical/Chemical Characterization Field Sheet (Form FD 9000-3):

- Submitting agency code
- Submitting agency name
- STORET station number
- Sample date
- Sample location including county
- Field identification
- Receiving body of water
- Time of sampling
- Percentage of land-use types in the watershed that drain to the site
- Potential for erosion within the portion of the watershed that affects the site
- Local non-point-source pollution potential and obvious sources
- Typical width of 100-meter section of river or stream
- Size of the system or the size of the sample area within the system (lake, wetland, or estuary)
- Three measurements of water depth across the typical width transect
- Three measurements of water velocity, one at each of the locations where water depth was measured
- Vegetated riparian buffer zone width on each side of the stream or river or at the least buffered point of the lake, wetland or estuary
- Presence of artificial channelization in the vicinity of the sampling location (stream or river)
- Description of state of recovery from artificial channelization
- Presence or absence of impoundments in the area of the sampling location
- Vertical distance from the current water level to the peak overflow level
- Distance of the high water mark above the stream bed
- Observed water depth at high water mark location
- Percentage range that best describes the degree of shading in the sampling area
- Any odors associated with the bottom sediments
- Presence or absence of oils in the sediment
- Any deposits in the area, including the degree of smothering by sand or silt
- Depth of each water quality measurement
- Temperature

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- pH
- Dissolved oxygen
- Specific conductance
- Salinity
- Secchi depth
- Type of aquatic system sampled
- Stream magnitude (order designation)
- Description of any noticeable water odors
- Term that best describes the TD
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- Term that best describes the TD
7/20/11 of turbidity in the water
- Term that best describes the color of the water
- Weather conditions during the time of sampling
- Any other conditions/observations that are helpful in characterizing the site
- Relative abundances of periphyton, fish, aquatic macrophytes and iron/sulfur bacteria
- List and map of dominant vegetation observed
- Sampling team designation
- Signature(s) of sampler(s)
- Signature date

2. For streams and rivers, draw a grid sketch of the site (optionally use Form FD 9000-4), showing the location and amount of each substrate type (as observed by sight or touch). Using the grid sketch, count the number of grid spaces for each substrate type. Divide each of these numbers by the total number of grid spaces contained within the site sketch. Record this percent coverage value for each substrate type. If the substrates are sampled, record the number of times each substrate is sampled by an indicated method.

3. For lakes, divide the site map into twelve sections and note visual markers that will assist in distinguishing those sections.

4. Photographs of the sampling area are also useful tools for documenting habitat conditions and identifying station location.

FD 5312. *Stream and River Biological Habitat Assessment Records (FT 3100)*

1. Record the following information or use Form FD 9000-5, Stream/River Habitat Assessment Field Sheet:

- Submitting organization name and/or code
- STORET station number
- Assessment date
- Sampling location including county
- Field identification

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- Receiving body of water
 - Time of sampling upon arrival at the site
2. Additionally record the following:
- Substrate diversity score
 - Substrate availability score
 - Water velocity s TD
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 - Habitat smothering score
 - Artificial channelization score
 - Bank stability score for each bank
 - Riparian buffer zone width score for each bank
 - Riparian zone vegetation quality score for each bank
 - Primary habitat components score
 - Secondary habitat components score
 - Habitat assessment total score
 - Additional comments and observations
 - Signatures
3. Record the following information or use Form FD 9000-4, Stream/River Habitat Sketch Sheet for each 100-meter segment assessed.
- Link to the waterbody name, location of 100-meter segment, analyst name(s) and date of the assessment
 - Code, symbol or icon used to map each substrate observed in the segment
 - Proportionate sketch or map of the abundance of each habitat (substrate) observed in the 100-meter segment, oriented to the direction of flow
 - Location of velocity measurements taken within the segment
 - Location of habitats smothered by sand or silt
 - Location of unstable, eroding banks
 - Locations along the segment where the natural, riparian vegetation is altered or eliminated
 - Plant taxa observed
 - Additional notes and observations

FD 5313. *Lake Biological Habitat Assessment Records (FT 3200)*

1. Document the following information or use the Lake Habitat Assessment Field Sheet (Form FD 9000-6):

- STORET station number

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- Sampling date
- Sampling location including lake name
- Eco-region
- Field identification number
- County name
- Lake size
- Features observed
- Description of the hydrology of the system (water residence time)
- Lake water color
- Secchi depth score
- Vegetation quality score
- Stormwater inputs score
- Bottom substrate quality score
- Lakeside adverse human alterations score
- Upland buffer zone score
- Adverse watershed land use score
- Habitat assessment total score
- Additional comments and observations
- Name and Signature of analyst

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FD 5320. BIOLOGICAL AQUATIC COMMUNITY SAMPLING RECORDS (FS 7000)

Minimum documentation required for biological sampling for procedures described in FS 7000 is listed below according to requirements as specified in the indicated sampling DEP SOPs.

FD 5321. *Periphyton Sampling Records (FS 7200)*

For each sample, record the following:

- Station sampled
- Date collected

FD 5322. *Qualitative Periphyton Sampling Records (FS 7220)*

Complete the Physical/Chemical Characterization Field Sheet (Form FD 9000-3), Stream/River Habitat Sketch Sheet (Form FD 9000-4) or site map and Stream/River Habitat Assessment Field Sheet (Form FD 9000-5), as appropriate for the water body sampled (see FT 3000 – FT 3100). Other customized formats may be used to record the information prompted on the above forms.

FD 5323. *Rapid Periphyton Survey Records (FS 7230)*

For each 100-meter reach surveyed, record the following information or use Form FD 9000-8, Rapid Periphyton Survey Field Sheet:

- Site or waterbody name
- Survey date
- Name(s) of analyst(s)
- Transect mark number (10-meter segment within the 100-meter reach)
- Transect point (1 – 9)
- Algae sample collection TD
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- Algal thickness rating (per FS 7230 procedure)
- Algae type
- Canopy cover (per FS 7230 procedure)
- Bottom visibility
- Water color
- Additional comments or observations

FD 5324. *Lake Vegetation Index Records (FS 7310)*

Record the following information or use Form FD 9000-7, Lake Vegetation Index Data Field Sheet:

- Waterbody name
- Assessment or sampling date
- County name
- Name of analyst(s)
- STORET station number
- Signature(s) of analyst(s)
- Lake water level
- Presence of algal mats
- Lake units sampled (12-sector procedure per FS 7310)
- Taxa observed in each selected unit
- Dominant and co-dominant taxa in each unit
- Taxa collected for further identification
- Approximate water depth for each taxon collected

FD 5325. *Rapid Bioassessment (Biorecon) Records (FS 7410)*

Record the following information or use the Biorecon Field Sheet (Form FD 9000-1).

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- STORET station number
- Location, including latitude and longitude
- Watershed or basin name
- Family or genus of all organisms from all material in all four dipnet sweeps
- Total taxa tallies
- Taxa richness, Ephemeroptera taxa, Trichoptera taxa, Long-lived taxa, Clinger taxa, and Sensitive taxa
- Abundance code for each taxon
- Name(s) of analyst(s) collecting and sorting samples
- Habitat types (subsampled) TD
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- Name(s) of analyst(s) performing quality control
- Signatures
- Collection date and time

FD 5326. *Stream Condition Index (D-frame Dipnet) Sampling Records (FS 7420)*

1. Complete the Physical/Chemical Characterization Field Sheet (Form FD 9000-3), Stream/River Habitat Sketch Sheet (Form FD 9000-4) or site map and Stream/River Habitat Assessment Field Sheet (Form FD 9000-5) forms appropriate for the water body sampled (see FT 3000 – FT 3400). Other customized formats may be used to record the information prompted on the above forms.
2. Record the following for each sample:
 - Number of sweeps for each habitat
 - Number of containers per sample

FD 5327. *Sediment Core Biological Grab Sampling Records (FS 7440)*

Record the sampling location of site grab core samples.

FD 5328. *Sediment Dredge Biological Grab Sampling Records (FS 7450)*

Record the sampling location of site grab dredge samples.

FD 5329. *Lake Condition Index (Lake Composite) Sediment Dredge Biological Grab Sampling Records (FS 7460)*

Record the following or use DEP Form FD 9000-2 (Composite Lake Sampling Sheet):

- Sampling date
- Lake name
- Sampling equipment used
- Comments and observations

- Dredge drop number (1 – 12)
- Sampling depth for each drop number
- Sampling location of site (include lake sector map)
- Sediment type(s) in grab dredge sample for each drop
- Location of any water quality measurements

FD 6000. QUALITY CONTROL DOCUMENTATION

1. Document all field quality control samples in the permanent field records.
2. At a minimum, record the following information:
 - The type, time and date that the quality control sample was collected; and
 - The preservative(s) (premeasured or added amount) and preservation checks performed.
3. If blanks are collected/prepared by the field organization, maintain records of the following:
 - Type of analyte-free water used;
 - Source of analyte-free water (include lot number if commercially purchased);
 - A list of the sampling equipment used to prepare the blank.

If items above are specified in an internal SOP, you may reference the SOP number and revision date in the field notes. Note any deviations to the procedure in the field notes.

4. For trip blanks, record the following:
 - Date and time of preparation
 - Storage conditions prior to release to the sample collecting organization
 - Type of analyte-free water used
 - Source and lot number (if applicable) of analyte-free water
 - 4.1. Include trip blank information in the sampling kit documentation per FD 2000, section 2.
5. For duplicates, record the technique that was used to collect the sample.
6. For split samples, identify the method used to collect the samples and the source(s) of the sample containers and preservatives.

FD 7000. LEGAL OR EVIDENTIARY DOCUMENTATION

1. Scope: The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance, for example, unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.
2. General Procedural Instructions
 - 2.1. Follow applicable requirements in FD 1000 – FD 5000 for all evidence samples.

2.2. Establish and maintain the evidentiary integrity of samples and/or sample containers. Demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

2.2.1. Document and track all time periods and the physical possession and storage of sample containers and samples from point of origin through the final analytical result and sample disposal.

FD 7100. General Requirements for Evidentiary Documentation

1. CHAIN OF CUSTODY RECORDS: Use the Chain-of-Custody (COC) records to establish an intact, contiguous record of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items are referred to as "samples".

1.1. Account for all time periods associated with the physical samples.

1.2. Include signatures of all individuals who physically handle the samples.

1.2.1. The signature of any individual on any record that is designated as part of the Chain-of-Custody is their assertion that they personally handled or processed the samples identified on the record.

1.2.2. Denote each signature with a short statement that describes the activity of the signatory (e.g., "sampled by", "received by", "relinquished by", etc.).

1.2.3. In order to simplify recordkeeping, minimize the number of people who physically handle the samples.

2. CONSOLIDATION OF RECORDS: The COC records need not be limited to a single form or document. However, limit the number of documents required to establish COC, where practical, by grouping information for related activities in a single record. For example, a sample transmittal form may contain both certain field information and the necessary transfer information and signatures for establishing delivery and receipt at the laboratory.

3. LIABILITY FOR CUSTODY DOCUMENTATION: Ensure appropriate personnel initiate and maintain sample chain-of-custody at specified times.

3.1. Begin legal chain-of-custody when the precleaned sample containers are dispatched to the field.

3.1.1. Omit the transmittal record for precleaned sample containers if the same party provides the containers and collects the samples.

3.2. Sign the COC record upon relinquishing the prepared sample kits or containers.

3.3. Sign the COC record upon receipt of the sample kits or containers.

3.4. Thereafter, ensure that all parties handling the samples maintain sample custody (i.e., relinquishing and receiving) and documentation until the samples or sampling kits are relinquished to a common carrier.

3.4.1. The common carrier should not sign COC forms.

3.4.2. Indicate the name of the common carrier in the COC record, when used. Retain shipping bills and related documents as part of the record.

3.4.3. Ensure that all other transferors and transferees releasing or accepting materials from the common carrier sign the custody record.

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3.5. Chain-of-custody is relinquished by the party who seals the shipping container and is accepted by the party who opens it.

3.5.1. Indicate the date and time of sealing of the transport container for shipment.

3.5.2. See FD 7200, section 3 below regarding the use of custody seals.

4. SAMPLE SHIPPING OR TRANSPORTING

4.1. Affix tamper-indicating custody seals or evidence tape before shipping samples.

4.1.1. Seal sample container caps with tamper-indicating custody seals or evidence tape before packing for shipping or transport.

4.1.2. Seal sample transport or shipping containers with strapping tape and tamper-indicating custody seals or evidence tape.

4.1.3. If the same party collects then possesses (or securely stores), packs and transports the samples from time of collection, omit any use of custody seals or evidence tape.

4.2. Keep the COC forms with the samples during transport or shipment. Place the COC records in a waterproof closure inside the sealed ice chest or shipping container.

FD 7200. Required Documentation for Evidentiary Custody

1. GENERAL CONTENT REQUIREMENTS: Document the following in COC tracking records by direct entry or linkage to other records:

- Time of day and calendar date of each transfer or handling procedure
- Signatures of transferors, transferees and other personnel handling samples
- Location of samples (if stored in a secured area)
- Description of all handling procedures performed on the samples for each time and date entry recorded above
- Storage conditions for the samples, including chemical preservation and refrigeration or other cooling
- Unique identification for all samples
- Final disposition of the physical samples
- Common carrier identity and related shipping documents

2. DOCUMENTATION CONTENT FOR SAMPLE TRANSMITTAL

Provide a Chain-of-Custody record for all evidentiary samples and subsamples that are transmitted or received by any party. Include the following information in the COC record of transmittal:

- Sampling site name and address
- Date and time of sample collection
- Unique field identification code for each sample source and each sample container
- Names of personnel collecting samples
- Signatures of all transferors and transferees

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- Time of day and calendar date of all custody transfers
- Clear indication of number of sample containers
- Required analyses by approved method number or other description
- Common carrier usage
- Sample container/preservation kit documentation, if applicable

3. CHAIN-OF-CUSTODY SEALS: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

- 3.1. Place the seal so that the closure cannot be opened without breaking the seal.
- 3.2. Record the time, calendar date, and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.
- 3.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FD 7300. Documenting Controlled Access to Evidence Samples

Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times. See FS 1000 for additional discussion about procedures for handling evidence samples.

1. Limit the number of individuals who physically handle the samples as much as practicable.
2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.
3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.
4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals.

FD 7400. Documenting Disposal of Evidence Samples

1. Dispose of the physical samples only with the concurrence of the affected legal authority, sample data user, and/or submitter/owner of the samples.
2. Record all conditions of disposal and retain correspondence between all parties concerning the final disposition of the physical samples.
3. Record the date of disposal, the nature of disposal (i.e., sample depleted, sample flushed into sewer, sample returned to client, etc.), and the name of the individual who performed the disposal. If samples are transferred to another party, document custody transfer in the same manner as other transfers (see FD 7000 – FD 7200).

FD 8000. (RESERVED)

FD 9000. FORMS

Forms to facilitate documentation of sampling, field-testing, and biological laboratory calculation activities are available on the Department's website. These forms are for unrestricted public use and are presented in example formats. *The use of these forms is not mandatory. However, **some** of the data elements and other information denoted by the form prompts comprise **required documentation** items. Not all required documentation is illustrated in the form examples.* Customize these forms as needed. These forms are available as separate document files. The following forms are incorporated into the indicated SOPs for convenience of use:

- Form FD 9000-1 Biorecon Field Sheet (FS 7000)
- Form FD 9000-2 Composite Lake Sampling Sheet for <1000 Acres (FS 7000)
- Form FD 9000-3 Physical/Chemical Characterization Field Sheet (FT 3000)
- Form FD 9000-4 Stream/River Habitat Sketch Sheet (FT 3000)
- Form FD 9000-5 Stream/River Habitat Assessment Field Sheet (FT 3000)
- Form FD 9000-6 Lake Habitat Assessment Field Sheet (FT 3000)
- Form FD 9000-7 Lake Vegetation Index Data Field Sheet (FS 7000)
- Form FD 9000-8 Rapid Periphyton Survey Field Sheet (FS 7000)

FM 1000. FIELD PLANNING AND MOBILIZATION

This SOP is advisory; however, the following procedures are designed as best management practices, for use as guidance for designing and implementing a field sampling program and when selecting a laboratory.

FM 2000. LABORATORY SCHEDULING

FM 2100. Selecting a Laboratory

1. CONSUMER RESPONSIBILITIES

Each organization that uses laboratory services has certain responsibilities to ensure that the laboratory has the appropriate credentials and that the data are useable for the intended needs, and acceptable to DEP. A consumer's responsibilities include:

1.1. Evaluating the Laboratory

1.1.1. Ensure that the laboratory has the proper credentials.

1.1.2. Ensure that the laboratory can produce data of a quality that will be acceptable to DEP.

1.2. Thinking in Terms of Quality not Dollars: A laboratory that produces data that are not acceptable to DEP usually means that the laboratory will need to repeat the work. It is more cost effective to select a laboratory that will meet the quality needs of the project even if that laboratory is not the lowest bidder.

1.3. Continuing Evaluation: In order to ensure that the laboratory provides data of a consistent quality, do not rely on just the initial evaluation of a laboratory. Other quality control measures will provide the ability to continuously evaluate the laboratory data quality.

1.4. Evaluating the Reported Data: Review the final laboratory reports against the original expectations and acceptable quality control measures.

1.5. Asking Questions: The consumer has the right to question laboratory results and receive a logical and clear response.

An informed client increases the probability of quality data and data acceptability.

FM 2110. IDENTIFYING LABORATORY NEEDS

The consumer should be able to identify these critical needs before considering any laboratory:

1. The purpose for which the data are needed.

1.1. The consumer must determine DEP's expectations for data quality in terms of the precision, accuracy, and detection limit (reporting level or criteria) for each reported value.

1.2. Examples include: permit compliance at some specified concentration levels; compliance monitoring at specified reporting levels; and site cleanup to specified soil and water criteria levels.

2. The benefits of using contracted or in-house analytical services.

3. The specific laboratory services that are required:

- 3.1. Are sample collection and sample analysis required, or just sample analysis.
- 3.2. Types of samples (groundwater, drinking water, soils, sediments, hazardous wastes, etc.).
- 3.3. The sample delivery schedule including:
 - 3.3.1. The number of samples to be collected.
 - 3.3.2. The frequency with which samples will be submitted to the laboratory.
 - 3.3.3. The types of matrices to be analyzed.
- 3.4. The test methods that must be used (normally found in the permit requirements, consent orders, contracts, or relevant rules).
- 3.5. The expected quality based on DEP's requirements.
- 3.6. The expected turnaround time for laboratory analysis.
- 3.7. The deliverables including the report format.
- 3.8. Field related services such as:
 - 3.8.1. Sample collection
 - 3.8.2. Sample containers
 - 3.8.3. Sample preservation
 - 3.8.4. Equipment rental or cleaning services; or
 - 3.8.5. Instrument calibration services.
4. Any required laboratory credentials such as certification.
5. Identifying key personnel in the consumer's organization that will be interfacing with the laboratory:
 - 5.1. Administrative contact: Usually responsible for obtaining laboratory services.
 - 5.2. Technical contact: Usually a person who will be evaluating the laboratory's performance.
 - 5.3. Sample control contact: Usually a person who will be scheduling services with the laboratory.
6. Have an understanding of the current market price for the tests to be performed.
 - 6.1. Gather information on pricing from several laboratories.
 - 6.2. Request current and historical pricing schedules.

FM 2120. EVALUATING THE LABORATORY

1. LABORATORY CREDENTIALS
 - 1.1. The laboratory must hold National Environmental Laboratory Accreditation Program (NELAP) certification from the Florida Department of Health's Environmental Laboratory Certification Program (DoH ELCP).
 - 1.2. Out-of-state laboratories must be either certified by DoH, or be NELAP-certified by another state **with secondary accreditation** by DoH.

- 1.3. The laboratory must be certified for the test technology, analyte, and matrices that will be requested. This does not apply to analysis being done for drinking water.
- 1.4. Request a copy of the Current Certification and Analyte Sheets (must be for the current fiscal year which runs July 1 to June 30).
- 1.5. Verify the certification through the DEP Web Site, or the DoH offices.

2. ON-SITE VISIT

Conduct an on-site visit to verify the laboratory's capabilities and to determine if the laboratory has the equipment and personnel resources necessary for proposed services.

- 2.1. The laboratory must show a willingness to meet the client's needs.
- 2.2. The laboratory (both the analytical and administrative areas) should appear organized.
- 2.3. The analytical staff must be knowledgeable about the services to be provided.
 - 2.3.1. At least one person (supervisor or analyst) must be experienced in performing all activities on the proposed scope of work.
- 2.4. The administrative staff must appear organized.
- 2.5. The laboratory must have the capacity to accommodate the proposed scope of work in terms of personnel and equipment.

3. LABORATORY PERFORMANCE EVALUATION

- 3.1. Blind Check Samples: Prior to contract signing or any agreement, submit a set of blind check samples to the laboratory.
 - 3.1.1. A blind check sample is a sample in a real matrix (water, soil, sediment, etc.) that appears to be a real sample, except that the submitter has a list of the components and their known concentration values.
 - 3.1.2. Submit the sample(s) to the laboratory as a routine sample(s).
 - 3.1.3. Evaluate the results of the reported values against the certified values in the sample(s).
 - 3.1.4. The values must be within the laboratory's stated precision for the measurement.

4. CUSTOMER SATISFACTION

- 4.1. Obtain a list of current and previous clients.
- 4.2. Call several of the clients to determine:
 - Satisfaction with laboratory
 - Were problems resolved satisfactorily?
 - Reasons for not using the laboratory (if applicable)
 - Reasons for using the laboratory

5. FISCAL STABILITY

- 5.1. Request a copy of the current financial statement.

FM 2130. CONTRACTING

1. PURPOSE
 - 1.1. Provide a detailed list of the scope of services to be contracted.
 - 1.2. Include the purpose for which the data are to be used (permit, compliance, etc.).
2. KEY CONTACTS: Identify key contacts for both laboratory and client:
 - 2.1. Administrative: Dealing with billing, contract writing, invoicing, etc.
 - 2.2. Technical: Dealing with data, and quality control issues and problems.
 - 2.3. Sample Control: Dealing with scheduling, shipping supplies, sample receipt.
3. ANTICIPATED NEEDS: Specify:
 - 3.1. The schedule of activities;
 - 3.2. The expected number of samples, analytes, matrices and tests; and
 - 3.3. Field support services, including containers, preservatives, cleaning and calibration services.
4. EXPECTATIONS
 - 4.1. Certification
 - 4.1.1. The laboratory must maintain certification for the analyte, technology, and matrices to be performed.
 - 4.1.2. The laboratory must immediately notify its clients if the certification status for any analyte changes.
 - 4.1.3. The laboratory must state that it will generate all results in strict compliance with the National Environmental Laboratory Accreditation Conference (NELAC) Standards.
 - 4.1.4. The laboratory must flag and justify any results that were not generated in accordance with NELAC.
 - 4.2. Analytical Expectations
 - 4.2.1. Provide a list of analytical methods to be performed and the matrices for each method.
 - 4.2.2. Provide a copy of the permit, QAPP, Sampling Plan or other document that outlines DEP's requirements.
 - 4.2.3. Specify the expected turn-around time for the analyses.
 - 4.2.4. Specify the shipping schedule if sample containers or supplies are to be provided.
 - 4.3. Container/Equipment Services: State the scope of container and equipment services:
 - 4.3.1. Precleaned Containers: Types and Numbers
 - 4.3.1.1. Must be cleaned according to DEP SOP procedures (see FC 1000) or purchased precleaned from a vendor.
 - 4.3.1.2. Provide copy of procedures, if the laboratory does not follow the DEP SOP procedures.

- 4.3.1.3. Determine if containers must be certified clean by either the laboratory or the vendor.
- 4.3.2. Preservatives
 - 4.3.2.1. Premeasured into containers, where appropriate.
 - 4.3.2.2. Provided in appropriate containers with dispensing implement.
- 4.3.3. Equipment
 - 4.3.3.1. Type and numbers.
 - 4.3.3.2. Condition of equipment (precleaned, etc.).
 - 4.3.3.3. Equipment must be cleaned according to DEP SOP procedures (see FC 1000). Obtain a copy of the laboratory procedures if the laboratory does not follow the DEP SOP procedures.
 - 4.3.3.4. Determine if equipment must be certified clean by the laboratory.
- 4.3.4. Equipment Calibration
 - 4.3.4.1. The calibration method;
 - 4.3.4.2. The frequency of calibration;
 - 4.3.4.3. Preventative maintenance on instrument;
 - 4.3.4.4. Certification statement verifying the calibration; and
 - 4.3.4.5. Documentation of all maintenance and calibrations in laboratory records.
- 4.4. Quality Control
 - 4.4.1. State adherence to NELAC quality control requirements.
 - 4.4.2. Specify any additional quality control measures that are required but are different from NELAC.
 - 4.4.3. Specify acceptable ranges for spikes, duplicates, surrogates, and other QC measures if appropriate.
- 4.5. Custody/Sample Tracking
 - 4.5.1. Specify adherence to NELAP documentation and record keeping requirements.
 - 4.5.2. State a time-period for retaining all records if greater than 5 years.
 - 4.5.3. Make arrangement for transfer of records should the laboratory go out of business or transfer ownership before the records retention time period has lapsed.
 - 4.5.4. Specify the level of custody (routine, legal, etc.).
- 4.6. Minimum Reporting Levels
 - 4.6.1. Provide the laboratory with the minimum acceptable values to be reported (method detection limit, etc.).
 - 4.6.2. Describe contingencies if these levels cannot be met.
- 4.7. Reporting Format
 - 4.7.1. All analytical reports issued by the laboratory must comply with DEP and NELAP reporting requirements.

4.7.2. Specify whether the information must be provided as hardcopy, electronic or both.

4.7.2.1. If electronic, specify the format for submission.

4.7.3. The use of appropriate DEP data qualifiers (see Table FM 1000-1) must be used.

4.8. Deliverables: In addition to the NELAP-compliant report, specify any other deliverables that must be provided with the laboratory report such as:

- Laboratory Quality Control results;
- Field Quality Control results;
- Performance Test results;
- Copies of all raw data and associated records;
- Written narrative of the analytical event; and/or
- Description of any modifications to methods.

4.9. Subcontracting

4.9.1. The laboratory must inform the client **before** any analytical services are subcontracted to another laboratory.

4.9.2. The laboratory must ensure that the subcontracted laboratory meets the same qualifications and requirements as the primary laboratory.

4.9.3. If the results from subcontracted laboratories are incorporated into the final laboratory report, the subcontracted results must be clearly identified.

4.10. Method Modifications

4.10.1. The laboratory must identify any modifications that have been made to the requested analytical methods.

4.10.2. The client must be notified of any method modifications prior to use in the laboratory, and must provide written consent.

4.11. Dilutions

4.11.1. Negotiate how multiple dilutions will be handled. They may be considered a separate analysis and therefore an additional cost.

4.11.2. Agree to pay for the analysis of dilutions only if:

4.11.2.1. The sample concentration exceeds the calibration range and the laboratory was not aware of the expected sample concentration; or

4.11.2.2. A dilution is required to quantitate all required components.

5. PENALTIES AND CONSEQUENCES

5.1. Negotiate penalties or other consequences (no payment) for these problems:

- Failure to provide data or associated (expected) information;
- Failure to meet deadlines;
- Failure to provide acceptable data; and
- Failure to meet contract requirements.

- 5.2. Consider these consequences:
 - Costs of resampling;
 - Fines incurred because of unacceptable data;
 - Costs associated with having evaluated and/or processed unacceptable data;
and
 - Reanalysis costs (if reanalysis is due to laboratory error or failed QC).
- 5.3. Reserve the right to reject data. If any data are used, laboratory should be paid according to negotiated terms.

FM 2140. ON-GOING EVALUATION

1. Monitor laboratory's performance against the specific contract requirements.
2. Continue to use blind QC samples as a measure of routine performance.
 - 2.1. Vendor supplied samples;
 - 2.2. Samples prepared to a known concentration; or
 - 2.3. Split samples with another laboratory.

FM 2150. DATA REVIEW

1. Review the data for logical trends:
 - 1.1. Are the reported concentrations different from the routine (expected) levels?
 - 1.2. Is the same value reported for the same analyte (except non detects) in the same set of samples or over a historical period of time?
 - 1.3. Do the parts add up to the total?
 - 1.3.1. Ortho phosphate must be less than total phosphate.
 - 1.3.2. Total nitrate-nitrite must be equal to nitrate plus nitrite.
 - 1.3.3. Total values must be greater than or equal to dissolved values.
 - 1.4. Are different but related analyses consistent?
 - 1.4.1. High turbidity and high total suspended solids.
 - 1.4.2. High turbidity and increased method detection limits for other tests.
 - 1.5. Do results indicate a sample collection problem?
 - 1.5.1. High dissolved oxygen in groundwater.
 - 1.5.2. High turbidity and elevated metals results.
 - 1.6. Are the QC check samples within acceptable ranges?
 - 1.6.1. Are the ranges reasonable?
 - 1.7. Are non-detects reported correctly (should be a value with a "U")?
 - 1.8. Over the history of laboratory use, were any QC problems reported?
 - 1.9. Is there any laboratory or field blank contamination?

1.10. Do the reports contain all required information?

FM 2160. ASK QUESTIONS

Ask questions if:

- There are problems associated with the data review.
- The QC check sample data are not acceptable.
- The laboratory consistently reports the same QC failure.
- The laboratory uses different methods than requested.
- The laboratory subcontracts analyses without notifying the client.
- The laboratory does not meet contract requirements.
- The laboratory misses holding times.
- The laboratory fails to provide requested resource(s) (containers, calibration, etc.) in a timely manner.
- There any doubts about the acceptability of the data.
- Detection limits are above the expected values and the laboratory provides no reasonable explanation.

FM 2200. Scheduling Services

1. Notify the laboratory about the analytical and equipment needs at least a week in advance of the actual sampling trip.

2. Even if the trip is routine (monthly, weekly, quarterly compliance sampling), provide the laboratory with a written request. Include:

- Number and types of samples to be collected;
- Test methods to be performed;
- Expectations for quality control acceptance criteria (if not already listed in a contract);
- Estimated numbers of each type of container;
- Required preservatives, including whether the laboratory will dispense premeasured quantities into the sample containers;
- Preservation supplies such as graduated, disposable pipets;
- Additional preservatives (even if the containers are prepreserved);
- Sampling equipment including material construction;
- Shipping containers;
- Forms (both courier and transmittal/custody forms);
- Any calibration services;
- Estimated time of delivery;
- Expected turn-around time;

- Special needs such as "requires legal chain of custody" or "requires 24-hour turn-around time";
- Data processing services (such as completing regulatory forms); and
- Expected contamination levels. This is important if a highly contaminated site is sampled.

FM 3000. TRIP PLANNING

1. Ensure that everyone involved with the event understands the purpose of the trip:
 - 1.1. Review the associated sampling plan, quality assurance project plan or permit requirements.
 - 1.2. Review the applicable safety plans and site files.
2. Determine the number of people that will be required to complete the sampling activities within the allotted time frame. For safety and efficiency, a field team should consist of at least two people.
3. Identify sampling team member(s) and schedule a meeting of the sampling team.
 - 3.1. Develop a detailed itinerary and schedule.
 - 3.1.1. Plan to sample from the least contaminated to the most contaminated sampling point.
 - 3.1.2. Plan to work upstream in flowing water.
 - 3.2. Review personnel training and make assignments based on experience.
 - 3.2.1. Ensure that at least one trained, experienced individual is part of the team.
 - 3.3. Review the SOPs and any associated documents (sampling plan, quality assurance project plan, permit, etc.).
 - 3.4. Review project/site files for unusual procedures or site peculiarities.
 - 3.5. Review the safety plan and discuss contingencies (weather, broken equipment, site access, etc.).
 - 3.5.1. If the sampling event is more than 3 - 5 days, a written contingency plan is recommended.
 - 3.5.2. If a boat will be used, a float plan is highly recommended.
 - 3.5.3. At a minimum discuss and have available:
 - 3.5.3.1. Phone and directions to nearest emergency facility;
 - 3.5.3.2. Phone number(s) of supervisor and/or project manager;
 - 3.5.3.3. Locations of power lines and underground utilities; and
 - 3.5.3.4. Expected environmental hazards.
4. Schedule the date for deployment and the duration of the sampling event.
 - 4.1. Obtain the necessary entry permits, keys, etc.
 - 4.2. Identify name(s) and phone number(s) of landowner, tenant or other responsible party.

5. Assemble any needed maps, directions and site descriptions. Include information on:
 - 5.1. Traffic conditions and/or traffic patterns; and
 - 5.2. Parking areas.
6. Identify the number of sampling points, and for each sampling point:
 - 6.1. Determine the matrices that will be sampled;
 - 6.2. Identify the specific analyses to be performed per matrix;
 - 6.3. Identify the sampling equipment needs based on the matrix and analytes to be collected. Include tubing, mixing implements and other support equipment;
 - 6.4. Based on the analytical tests and the matrices, determine the number and types of sample containers;
 - 6.5. Based on the analytical tests and the matrices, determine the types of preservatives that will be needed;
 - 6.6. Determine what field measurements must be made; and
 - 6.7. Identify transportation mode to reach the location (boat, truck, etc.).
7. Calculate the total number of each container types (both preserved and unpreserved).
8. Determine the total number of sampling equipment sets (tubing, mixing trays, coring devices, etc.) that will be needed for the sampling event.
9. Notify the laboratory of the trip and arrange for necessary containers, preservatives and other supplies (see FM 2200).
10. Reserve appropriate vehicles.
11. Assemble all field records (notebooks, forms, transmittal forms, etc.).

FM 4000. EQUIPMENT AND SUPPLY PREPARATION

1. SAMPLING EQUIPMENT: Assemble all equipment identified in FM 3000, section 8.
 - 1.1. Inspect equipment for cracks, breaks, and other signs of wear.
 - 1.2. If necessary, repair any equipment and document the repairs in appropriate maintenance logs.
 - 1.3. Reclean any equipment that was cleaned but not protected from the environment (stored on dusty shelves).
 - 1.3.1. If not already clean, decontaminate equipment according to FC 1000.
 - 1.3.2. Clean all transport ice chests and water transport containers (see FC 1190 and FC 1180, respectively).
 - 1.4. Check to make sure fuel and battery powered pumps are working.
 - 1.5. See "Field Sample Collection Equipment Checklist".
2. FIELD MEASUREMENTS: Assemble field instruments to make the measurements identified in FM 3000, section 6.6.
 - 2.1. Inspect instruments for damage.

- 2.1.1. Repair and/or replace parts as necessary, and document in appropriate maintenance logs.
 - 2.1.2. Assemble the appropriate calibration standards and supplies.
 - 2.1.3. Determine the accuracy of the instruments by either performing an initial calibration or checking the calibration before leaving the base of operations. Document the calibration check.
- 2.2. See "General Field Support Equipment Checklist", item 7.
3. DOCUMENTATION: Assemble field record supplies:
 - Notebooks, and/or forms
 - Indelible/waterproof pens
 - Clipboards
 - Cameras
 - GPS unit, if needed
 - See "General Field Support Equipment Checklist".
4. SAMPLE CONTAINERS: Assemble the appropriate types of sample containers or obtain them from the contracted laboratory. See "General Field Support Equipment Checklist", item 8.
5. PRESERVATIVES: Assemble preservation supplies if not provided by the laboratory.
 - 5.1. Discard any old solutions; clean containers; and prepare fresh solutions.
 - 5.2. See "General Field Support Equipment Checklist", item 2.
6. FIELD DECONTAMINATION SUPPLIES: Assemble field decontamination supplies.
 - 6.1. Discard any old solutions; clean containers; and prepare fresh solutions.
 - 6.2. Discard analyte-free water and obtain fresh water.
 - 6.3. See "General Field Support Equipment Checklist", item 1.
7. SHIPPING SUPPLIES: Assemble shipping supplies:
 - 7.1. Determine nearest point to obtain ice;
 - 7.2. Marking pens, shipping labels, tape, custody seals (if required);
 - 7.3. See "General Field Support Equipment Checklist", item 3.
8. VEHICLES:
 - 8.1. Make sure vehicle maintenance is up-to-date.
 - 8.2. Check fluids.
 - 8.3. Check tire pressure.
 - 8.4. Check fuel and fuel supply.
 - 8.5. See "General Field Support Equipment Checklist", item 10.

9. SAFETY EQUIPMENT: Assemble any needed safety equipment:
- Protective gloves.
 - Protective clothing including boots.
 - SCUBA gear or other supplied air supply.
 - First aid kit.
 - Drinking water.
 - Float plan.
 - Address and phone numbers for nearest emergency room.
 - See "General Field Support Equipment Checklist", item 6.

Appendix FM 1000

Tables, Figures and Checklists

Table FM 1000-1 Data Qualifier Codes

General Field Support Equipment Checklist

Field Sample Collection Equipment Checklist

**Table FM 1000-1
 DATA QUALIFIER CODES**

The following codes shall be used by laboratories and/or field organizations when reporting data values that either meet the specified description outlined below or do not meet the quality control criteria of the laboratory:

Symbol	Meaning
A	Value reported is the arithmetic mean (average) of two or more determinations. This code shall be used if the reported value is the average of results for two or more discrete and separate samples. These samples shall have been processed and analyzed independently. Do not use this code if the data are the result of replicate analysis on the same sample aliquot, extract or digestate.
B	Results based upon colony counts outside the acceptable range. This code applies to microbiological tests and specifically to membrane filter colony counts. The code is to be used if the colony count is generated from a plate in which the total number of coliform colonies is outside the method indicated ideal range. This code is not to be used if a 100 mL sample has been filtered and the colony count is less than the lower value of the ideal range.
F	When reporting species: F indicates the female sex.
H	Value based on field kit determination; results may not be accurate. This code shall be used if a field screening test (i.e., field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
I	The reported value is greater than or equal to the laboratory method detection limit but less than the laboratory practical quantitation limit.
J	Estimated value. A "J" value shall be accompanied by a detailed explanation to justify the reason(s) for designating the value as estimated. Where possible, the organization shall report whether the actual value is estimated to be less than or greater than the reported value. A "J" value shall not be used as a substitute for K, L, M, T, V, or Y, however, if additional reasons exist for identifying the value as an estimate (e.g., matrix spiked failed to meet acceptance criteria), the "J" code may be added to a K, L, M, T, V, or Y. Examples of situations in which a "J" code must be reported include: instances where a quality control item associated with the reported value failed to meet the established quality control criteria (the specific failure must be identified); instances when the sample matrix interfered with the ability to make any accurate determination; instances when data are questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of a grab sample); instances when the analyte was detected at or above the method detection limit in a blank other than the method blank (such as calibration blank or field-generated blanks and the value of 10 times the blank value was equal to or greater than the associated sample value); or instances when the field or laboratory calibrations or calibration verifications did not meet calibration acceptance criteria.

**Table FM 1000-1
 DATA QUALIFIER CODES**

Symbol	Meaning
K	Off-scale low. Actual value is known to be less than the value given. This code shall be used if:
	1. The value is less than the lowest calibration standard and the calibration curve is known to be non-linear; or
	2. The value is known to be less than the reported value based on sample size, dilution.
	This code shall not be used to report values that are less than the laboratory practical quantitation limit or laboratory method detection limit.
L	Off-scale high. Actual value is known to be greater than value given. To be used when the concentration of the analyte is above the acceptable level for quantitation (exceeds the linear range or highest calibration standard) and the calibration curve is known to exhibit a negative deflection.
M	When reporting chemical analyses: presence of material is verified but not quantified; the actual value is less than the value given. The reported value shall be the laboratory practical quantitation limit. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is greater than <u>or equal to</u> the method detection limit. If the value is less than the method detection limit use "T" below.
N	Presumptive evidence of presence of material. This qualifier shall be used if:
	1. The component has been tentatively identified based on mass spectral library search; or 2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternative procedures).
O	Sampled, but analysis lost or not performed.
Q	Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared or analyzed after the approved holding time restrictions for sample preparation or analysis.
T	Value reported is less than the laboratory method detection limit. The value is reported for informational purposes only and shall not be used in statistical analysis.
U	Indicates that the compound was analyzed for but not detected. This symbol shall be used to indicate that the specified component was not detected. The value associated with the qualifier shall be the laboratory method detection limit. Unless requested by the client, less than the method detection limit values shall not be reported (see "T" above).
V	Indicates that the analyte was detected at or above the method detection limit in both the sample and the associated method blank and the value of 10 times the blank value was equal to or greater than the associated sample value. Note:

**Table FM 1000-1
 DATA QUALIFIER CODES**

Symbol	Meaning
	unless specified by the method, the value in the blank shall not be subtracted from associated samples.
X	Indicates, when reporting results from a Stream Condition Index Analysis (LT 7200 and FS 7420), that insufficient individuals were present in the sample to achieve a minimum of 280 organisms for identification (the method calls for two aliquots of 140-160 organisms), suggesting either extreme environmental stress or a sampling error.
Y	The laboratory analysis was from an improperly preserved sample. The data may not be accurate.
Z	Too many colonies were present for accurate counting. Historically, this condition has been reported as "too numerous to count" (TNTC). The "Z" qualifier code shall be reported when the total number of colonies of all types is more than 200 in all dilutions of the sample. When applicable to the observed test results, a numeric value for the colony count for the microorganism tested shall be estimated from the highest dilution factor (smallest sample volume) used for the test and reported with the qualifier code.
?	Data are rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
*	Not reported due to interference.

The following codes deal with certain aspects of field activities. The codes shall be used if the laboratory has knowledge of the specific sampling event. The codes shall be added by the organization collecting samples if they apply:

Symbol	Meaning
D	Measurement was made in the field (i.e., in situ). This <u>code</u> applies to any value (except <u>field measurements of pH, specific conductance, dissolved oxygen, temperature, total residual chlorine, transparency, turbidity or salinity</u>) that was obtained under field conditions using approved analytical methods. If the parameter code specifies a field measurement (e.g., "Field pH"), this code is not required.
E	Indicates that extra samples were taken at composite stations.
R	Significant rain in the past 48 hours. (Significant rain typically involves rain in excess of 1/2 inch within the past 48 hours.) This code shall be used when the rainfall might contribute to a lower than normal value.
!	Data deviate from historically established concentration ranges.

General Field Support Equipment Checklist

Date: _____	Project/Site: _____	
DECONTAMINATION SUPPLIES <input type="checkbox"/> Basins, buckets or bowls to hold wash water and various rinse waters <input type="checkbox"/> Brushes or other implements to clean equipment <input type="checkbox"/> Detergents <input type="checkbox"/> Liqui-Nox or equivalent <input type="checkbox"/> Alconox or equivalent <input type="checkbox"/> Acids <input type="checkbox"/> Nitric <input type="checkbox"/> Hydrochloric <input type="checkbox"/> Solvents <input type="checkbox"/> Pesticide grade isopropanol <input type="checkbox"/> Other: _____ <hr/> <input type="checkbox"/> Protective wrapping <input type="checkbox"/> Foil <input type="checkbox"/> Untreated Plastic bags <input type="checkbox"/> Bubble wrap <input type="checkbox"/> Analyte-free water <input type="checkbox"/> Distilled in HDPE <input type="checkbox"/> Deionized in HDPE <input type="checkbox"/> Organic-free in HDPE, Teflon or glass <input type="checkbox"/> Dispensing bottles <input type="checkbox"/> HDPE for acids and detergents <input type="checkbox"/> Teflon for solvents and organic-free water <input type="checkbox"/> Paper towels or other absorbent material <input type="checkbox"/> Containers for IDW PRESERVATION SUPPLIES <input type="checkbox"/> Acids <input type="checkbox"/> Nitric <input type="checkbox"/> Hydrochloric <input type="checkbox"/> Sulfuric <input type="checkbox"/> Dechlorination reagents <input type="checkbox"/> Sodium thiosulfate <input type="checkbox"/> Ascorbic acid <input type="checkbox"/> Sodium hydroxide <input type="checkbox"/> Dispensing devices	<input type="checkbox"/> Graduated disposable plastic pipets <input type="checkbox"/> Glass Pasteur pipets <input type="checkbox"/> Bulbs <input type="checkbox"/> Premeasured reagents in vials <input type="checkbox"/> Narrow range pH paper (range of no more than 3 pH units) <input type="checkbox"/> pH range of 1 – 3 <input type="checkbox"/> pH range of 11 – 14 <input type="checkbox"/> pH range of 6 – 8 <input type="checkbox"/> Cyanide processing <input type="checkbox"/> Sulfide test paper <input type="checkbox"/> Precipitating Chemical <input type="checkbox"/> Cadmium nitrate or <input type="checkbox"/> Cadmium carbonate or <input type="checkbox"/> Lead nitrate or <input type="checkbox"/> Lead carbonate <input type="checkbox"/> KI starch paper <input type="checkbox"/> Ascorbic acid <input type="checkbox"/> Filter paper SAMPLE TRANSPORTATION SUPPLIES <input type="checkbox"/> Ice chests <input type="checkbox"/> Wet ice <input type="checkbox"/> Sealing tape <input type="checkbox"/> Shipping labels <input type="checkbox"/> Shipping forms <input type="checkbox"/> Bubble wrap <input type="checkbox"/> Plastic bags <input type="checkbox"/> Vermiculite <input type="checkbox"/> Custody seals DOCUMENTATION SUPPLIES <input type="checkbox"/> Notebooks/logs/field forms <input type="checkbox"/> Pens and markers (waterproof) <input type="checkbox"/> Sample container labels/tags <input type="checkbox"/> Custody tags <input type="checkbox"/> Custody/transmittal forms <input type="checkbox"/> Clipboard <input type="checkbox"/> Camera <input type="checkbox"/> Film	<input type="checkbox"/> GPS equipment <input type="checkbox"/> Calculator REFERENCE MATERIALS <input type="checkbox"/> Site maps and directions <input type="checkbox"/> QAPP <input type="checkbox"/> Sampling plan <input type="checkbox"/> SOPs <input type="checkbox"/> Itinerary <input type="checkbox"/> Float plan <input type="checkbox"/> Contingency plan HEALTH & SAFETY SUPPLIES <input type="checkbox"/> Cell phone <input type="checkbox"/> First aid kit <input type="checkbox"/> Drinking water <input type="checkbox"/> Protective gloves <input type="checkbox"/> Insect repellent <input type="checkbox"/> Sunscreen <input type="checkbox"/> Numbers for nearest emergency facilities <input type="checkbox"/> Safety goggles <input type="checkbox"/> Applicable MSDS sheets <input type="checkbox"/> Respirators <input type="checkbox"/> Fire extinguisher <input type="checkbox"/> Hard hats <input type="checkbox"/> Flotation jackets <input type="checkbox"/> Cable cutters <input type="checkbox"/> Traffic cones <input type="checkbox"/> SCUBA gear <input type="checkbox"/> SCBA gear <input type="checkbox"/> Other personal protection gear FIELD MEASUREMENT EQUIPMENT <input type="checkbox"/> Lint-free tissues <input type="checkbox"/> Flow-through cells <input type="checkbox"/> pH meter <input type="checkbox"/> 4, 7 & 10 buffers <input type="checkbox"/> Conductivity meter <input type="checkbox"/> Solution at expected conductivity <input type="checkbox"/> DO meter <input type="checkbox"/> Turbidimeter <input type="checkbox"/> Gel or Formazin standards

General Field Support Equipment Checklist

Date: _____

Project/Site: _____

- Residual chlorine
 - Secondary or primary standards
- Secchi disk
- MultiProbe

SAMPLE CONTAINERS

- Extractable Organics
 - Volatile Organics
 - Nutrients
 - Glass
 - Plastic
 - Inorganic Non-metallics
 - Glass
 - Plastic
 - Physical Parameters
 - Glass
 - Plastic
 - Metals
 - Glass
 - Plastic
 - Microbiology
 - Glass
 - Plastic
 - Whole Effluent Toxicity
 - Tissues
 - Macrobenthic invertebrates
 - Periphyton
 - Sediment/Soil volatiles
 - Sediment/Soil
- Remember:
- Extra containers
 - Extra VOC septa

FILTRATION EQUIPMENT

- 1 µm filter units
- 0.45 µm filters
- Peristaltic pump
- Pressurized bailers
- Syringe with Luer-Lok fitting
- Tripod filter with pressure/vacuum source
- Forceps for handling filters

VEHICLES

- Truck
- Fuel
- Boat
- Fuel
- Motor
- Paddles/oars
- Safety vests

MISCELLANEOUS SUPPLIES

- Hip boots
- Chest waders
- Rain gear
- Tool kit
- Extra batteries
- Stopwatch

Field Sample Collection Equipment Checklist

Date: _____ Project/Site: _____

- GROUNDWATER**
- Pumps
- Peristaltic
 - Centrifugal
 - Variable speed submersible
 - Submersible
 - Variable speed bladder
 - Bladder
- Tubing
- Teflon _____ Sets
 - Polyethylene _____ Sets
 - Polypropylene _____ Sets
 - Vinyl _____ Sets
 - Rubber _____ Sets
 - Tygon _____ Sets
- Bailers
- Teflon TD
 - Stainless steel 7/20/11
 - Polyethylene 7/20/11
 - Acrylic
 - PVC
- Support Equipment
- Graduated containers for measuring purge water
 - Containers for holding purge waters
 - Water level measuring device
 - Plastic sheeting
 - Lanyard material
 - Reels
 - Energy source for pumps

- SURFACE WATER**
- Pumps:
- Peristaltic
 - Automatic sampler TD
 - Other 7/20/11
- Tubing
- Teflon™ _____ Sets
 - Polyethylene _____ Sets
 - Polypropylene _____ Sets
 - Vinyl _____ Sets
 - Rubber _____ Sets
 - Tygon _____ Sets

- Bailers
- Teflon TD
 - Stainless steel 7/20/11
 - Polyethylene
 - Acrylic
 - PVC
- Grab Sampling Devices:
- Dipper
 - Kemmerer
 - Alpha water sampler
 - Niskin
 - Beta sampler
 - Retrieval lines
- Mixing Implements
- Churn splitter

- WASTEWATER**
- Pond sampler
 - Dippers
 - Peristaltic pump
- Tubing
- Teflon TD _____ Sets
 - Polyethylene 7/20/11 _____ Sets
 - Polypropylene _____ Sets
 - Vinyl _____ Sets
 - Rubber _____ Sets
 - Tygon _____ Sets
 - Kemmerer
 - Van Dorn
 - Nansen
 - Alpha bottle
 - Beta bottle
 - Niskin
 - DO dunker
 - Automatic composite sampler
- Tubing
- Teflon _____ Sets
 - Polyethylene _____ Sets
 - Polypropylene _____ Sets
 - Vinyl _____ Sets
 - Rubber _____ Sets
 - Tygon _____ Sets
- Bailers
- Plastic
 - Teflon
 - Stainless steel

- Scoops
- Plastic
 - Teflon
 - Stainless steel
- Beakers
- Plastic
 - Teflon
 - Stainless steel
- Buckets
- Plastic TD
 - Stainless steel 7/20/11

- SEDIMENTS**
- Dredges
- Petersen
 - Ponar
 - Ekman
 - Young Grab
 - Van Veer
 - Shipek
 - Orange-peel grab
 - Smith-McIntyre grab
 - Drag buckets
 - Winch
 - Cable/line
 - Messenger
- Coring Devices
- Stainless steel
 - Glass
 - Plastic
 - Teflon-lined

- SOIL**
- Bucket auger
 - Split spoon sampler
 - Stainless steel shovel
 - Garden shovel
 - Stainless steel trowel or scoop
 - Plastic trowel or scoop
 - Trenching device
 - Coring Devices
 - Stainless steel
 - Glass
 - Plastic
 - Teflon-lined
 - Shelby tube
 - EnCore

Field Sample Collection Equipment Checklist

Date: _____ Project/Site: _____

- WASTE**
- Stainless steel scoop
 - Stainless steel spoons or spatulas
 - Stainless steel push tubes
 - Stainless steel auger
 - Stainless steel Ponar dredge
 - Glass coliwasa
 - Drum thief
 - Mucksucker
 - Dipstick
 - Stainless steel bacon bomb
 - Stainless steel bailer
 - Teflon bailer
 - Peristaltic pump
 - Stainless steel split spoon
 - Roto-hammer
 - Glass tubing
- SHELLFISH**
- Seine
 - Trawl
 - Bucket type/double pole
 - Tong/Double handed grab
 - Line or cable operated grab bucket
 - Petersen
 - Ponar
 - Ekman
 - Orange-peel grab
 - Biological or hydraulic dredge
 - Scoops/shovels
 - Scrapers
 - Rakes
 - D-traps
- Processing Equipment
- Holding trays
 - Stainless steel shucking knife
 - Calipers or ruler
 - Aluminum foil
 - Plastic bags
- FINFISH**
- Electrofishing devices
 - Seines

- Trawls
 - Angling
 - Gill net
 - Trammel net
 - Hoop, fyke & pound nets
 - D-traps
- Processing Equipment
- Holding trays
 - Measuring board or ruler
 - Stainless steel descaler
 - Stainless steel scalpel
 - Balance
 - Aluminum foil
 - Plastic bags
- BIOLOGICAL COMMUNITY SAMPLING**
- Phytoplankton
- Van Dorn
 - Alpha bottle
 - Logol's solution
- Periphyton
- Periphytometer
 - Microscope slides
 - 100% buffered formalin
 - Nylon twine
- Qualitative Periphyton Sampling
- Stainless steel spatula/spool
 - Stainless steel forceps
 - Suction bulb
 - Preservative
 - Buffered formalin
 - Lugol's solution
 - M3
 - Resealable plastic bags
 - White picking pan
- Benthic Macroinvertebrates
- Forceps
 - Transfer pipettes
 - White picking pans
 - 10X hand lens
 - Alcohol-filled jars
 - Dip net (30 mesh)
 - Hester-Dendy
 - Coring device

- Dredge
- Ekman
- Petite
- 30 mesh box sieve

TD
7/20/11

TD
7/20/11

TD
7/20/11

FQ 1000. FIELD QUALITY CONTROL REQUIREMENTS

Field quality control measures monitor the sampling event to ensure that the collected samples are representative of the sample source.

Field-collected blanks must demonstrate that the collected samples have not been contaminated by:

- The sampling environment
- The sampling equipment
- The sample container
- The sampling preservatives
- Sample transport
- Sample storage

FQ 1100. Sample Containers

Sample containers must be free from contamination by the analytes of interest or any interfering constituents and must be compatible with the sample type.

FQ 1200. Sampling Operations

1. When collected, analyze all quality control samples for the same parameters as the associated samples.

1.1. When collected, collect blanks for the following parameter groups and tests:

- Volatile Organics
- Extractable Organics
- Metals
- Ultratrace Metals
- Inorganic Nonmetallics
- Radionuclides
- Petroleum Hydrocarbons and Oil & Grease
- Volatile Inorganics
- Aggregate Organics except Biochemical Oxygen Demand

1.2. Blanks are not required for:

- Microbiological (all types)
- Toxicity
- Field parameters such as pH, Specific Conductance, Residual Chlorine, Temperature, Light Penetration, Dissolved Oxygen, ORP and Salinity
- Radon

- Algal Growth Potential
 - Biological Community
 - Physical and Aggregate Properties
 - Biochemical Oxygen Demand
2. Preserve, transport, document and handle all quality control samples as if they were samples. Once collected, they must remain with the sample set until the laboratory has received them.
 3. Except for trip blanks, prepare all quality control samples **on-site in the field**.
 - 3.1. Do not prepare precleaned equipment blanks in advance at the base of operations.
 - 3.2. Do not prepare field-cleaned equipment blanks after leaving the sampling site.
 4. Perform and document any field QC measures specified by the analytical method (such as trip blanks for volatile organics).

FQ 1210. QUALITY CONTROL BLANKS

FQ 1211. *Precleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil, or sediment samples.
2. Collect these blanks using sampling equipment that has been brought to the site precleaned and ready for use. The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.
3. Collect these blanks before the equipment set has been used.
4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).

FQ 1212. *Field-Cleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.
2. Collect these blanks using sampling equipment that has been cleaned in the field (i.e., between sampling points). The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.
3. Prepare field-cleaned equipment blanks immediately after the equipment is cleaned in the field and before leaving the sampling site.
4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).
 - 4.1. For intermediate sampling devices or equipment, site-water rinsing is defined as the decontamination step, if this is the only cleaning that will be performed on the equipment prior to collecting the sample.

- 4.1.1. In this case, collect the equipment blank after rinsing the intermediate device 3 times with site water
- 4.1.2. Follow the site-water rinses with 3 rinses using analyte-free water.
- 4.1.3. Collect the equipment blank with a subsequent rinse of the device using additional analyte-free water to collect sufficient blank volume.

FQ 1213. *Trip Blanks*

1. USE: Monitors sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.
2. The organization that is providing the VOC vials must provide the trip blanks by filling two or more VOC vials with analyte-free water and preservatives (if needed).
 - 2.1. To prevent degradation of the trip blank, long-term storage of prepared trip blanks is not recommended.
3. These blanks are applicable if samples are to be analyzed for volatile constituents (volatile organics, methyl mercury, etc.) in water, waste, soils, or sediments.
4. Place a set of trip blanks in each transport container used to ship/store empty VOC vials. They must remain with the VOC vials during the sampling episode and must be transported to the analyzing laboratory in the same shipping or transport container(s) as the VOC samples.
5. Trip blanks must be opened **only** by the laboratory after the blank and associated samples have been received for analysis.

FQ 1214. *Field Blanks*

1. USE: Monitors on-site sampling environment, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil or sediment samples.
2. Prepare field blanks by pouring analyte-free water into sample containers for each parameter set to be collected.
3. Field blanks are not required if equipment blanks (FQ 1211 or FQ 1212) are collected.

FQ 1220. **FIELD DUPLICATES**

1. USE: Designed to measure the variability in the sampling process.
2. GENERAL CONSIDERATIONS:
 - 2.1. Collect duplicates by **repeating** (simultaneously or in rapid succession) the entire sample acquisition technique that was used to obtain the first sample.
 - 2.1.1. Collect, preserve, transport and document duplicates in the same manner as the samples. **These samples are not considered laboratory duplicates.**
 - 2.2. When collected, analyze field duplicates for the same parameters as the associated samples.
 - 2.3. If possible, collect duplicate samples from sampling locations where contamination is present.

2.4. Field duplicates must be collected if required by the analytical method and as required by a DEP program.

FQ 1221. *Water Duplicates*

Collect water duplicates by sampling from successively collected volumes (i.e., samples from the next volume of sample water).

FQ 1222. *Soil Duplicates*

Collect soil duplicates from the same sample source (i.e., soil from the same soil sampling device).

FQ 1230. MANDATORY FIELD QUALITY CONTROLS

1. The respondent, permittee or contractor and the sampling organization are responsible for ensuring that blanks (excluding trip blanks) are collected at a minimum of 5% of each reported test result/matrix combination for the life of a project.

1.1. Collect at least one blank for each reported test result/matrix combination each year for each project.

1.2. If a party wishes to claim that a positive result is due to external contamination sources during sample collection, transport or analysis, then at least one field collected blank (excludes trip blanks) must have been collected at the same time the samples were collected and analyzed with the same sample set.

1.3. A project will be defined by the organization responsible for collecting the samples for the project.

1.3.1. When applicable, define the scope of the project in conjunction with the appropriate DEP authority.

2. When collecting a set of blanks, use the following criteria:

2.1. Equipment Blanks:

2.1.1. Collect field-cleaned equipment blanks if any sample equipment decontamination is performed in the field.

2.1.2. If no decontamination is performed in the field, collect precleaned equipment blanks if the equipment is not certified clean by the vendor or the laboratory providing the equipment.

2.1.3. Equipment blanks are not required for volatile organic compounds.

2.2. Field Blanks:

2.2.1. Collect field blanks if no equipment except the sample container is used to collect the samples or if the sampling equipment is certified clean by the vendor or the laboratory providing the equipment.

2.2.1.1. If a sample container is used as an intermediate sample collection device, collect an equipment blank by rinsing the decontaminated collection container as the substitute for the field blank.

2.2.2. Field blanks are not required for volatile organic compounds.

2.3. Trip Blanks:

2.3.1. These blanks are applicable if samples are to be analyzed for volatile organic compounds. See FQ 1213 for frequency, preparation and handling requirements.

3. OPTIONAL QUALITY CONTROL MEASURES

3.1. The method or project may require collection of additional quality control measures as outlined in FQ 1210 (Blanks), FQ 1220 (Duplicates) and FQ 1240 (Split Samples).

FQ 1240. SPLIT SAMPLES

The DEP or the client may require split samples as a means of determining compliance or as an added measure of quality control. Unlike duplicate samples that measure the variability of both the sample collection and laboratory procedures, split samples measure only the variability **between** laboratories. Therefore, the laboratory samples must be subsamples of the same parent sample and every attempt must be made to ensure sample homogeneity.

Collect, preserve, transport and document split samples using the same protocols as the related samples. In addition, attempt to use the same preservatives (if required).

If split samples are incorporated as an added quality control measure, the DEP recommends that all involved parties agree on the logistics of collecting the samples, the supplier(s) of the preservatives and containers, the analytical method(s), and the statistics that will be used to evaluate the data.

FQ 1241. Soils, Sediments, Chemical Wastes and Sludges

Collecting split samples for these matrices is not recommended because a true split sample in these matrices is not possible.

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FQ 1242. Water

Collect split samples for water in one of two ways:

1. Mix the sample in a large, appropriately precleaned, intermediate vessel (a churn splitter is recommended). This method shall not be used if volatile or extractable organics, oil and grease or total petroleum hydrocarbons are of interest. While continuing to thoroughly mix the sample, pour aliquots of the sample into the appropriate sample containers. Alternatively:

2. Fill the sample containers from consecutive sample volumes **from the same sampling device**. If the sampling device does not hold enough sample to fill the sample containers, use the following procedure:

2.1. Fill the first container with half of the sample, and pour the remaining sample into the second container.

2.2. Obtain an additional sample, pour the first half into the **second** container, and pour the remaining portion into the first container.

2.3. Continue with steps described in sections 2.1 and 2.2 above until both containers are filled.

FQ 1250. QUALITY CONTROL DOCUMENTATION

1. Document all field quality control samples in the permanent field records.
2. At a minimum, record the following information:

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FQ 1000 Field Quality Control Requirements

- The type, time and date that the quality control sample was collected; and
 - The preservative(s) (premeasured or added amount) and preservation checks performed.
3. If blanks are collected/prepared by the field organization, maintain records of the following:
- Type of analyte-free water used;
 - Source of analyte-free water (include lot number if commercially purchased);
 - A list of the sampling equipment used to prepare the blank.

If items above are specified in an internal SOP, you may reference the SOP number and revision date in the field notes. Note any deviations to the procedure in the field notes.

4. For trip blanks, record the following:
- Date and time of preparation
 - Storage conditions prior to release to the sample collecting organization
 - Type of analyte-free water used
 - Source and lot number (if applicable) of analyte-free water
- 4.1. Include trip blank information in the sampling kit documentation per FD 2000, section 2.
5. For duplicates, record the technique that was used to collect the sample.
6. For split samples, identify the method used to collect the samples and the source(s) of the sample containers and preservatives.

FS 2200. Groundwater Sampling

1. INTRODUCTION AND SCOPE

1.1 Use these Standard Operating Procedures to collect groundwater samples. They are designed to ensure that the collected samples will be representative of water in the aquifer or target formation and that the samples have not been altered or contaminated by the sampling and handling procedures. These procedures apply to permanently and temporarily installed monitoring wells, wells constructed using "direct-push" techniques, wells with installed plumbing, remedial groundwater treatment systems and excavations where groundwater is present. Use of alternative, DEP-approved and properly documented procedures (e.g., Corporate SOP, ASTM Standards, alternative equipment, etc.) is acceptable if they meet the intent (e.g., sample representativeness and integrity) of this standard (see FA 1000).

1.2 The topics in this SOP include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.3 Use the following DEP SOPs in conjunction with FS 2200:

- FA 1000 Regulatory Scope and Administrative Procedures for Use of DEP SOPs
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FS 2000 General Aqueous Sampling
- FT 1000 Field Testing and Measurement
- FT 1100 Field pH
- FT 1200 Field Specific Conductance
- FT 1400 Field Temperature
- FT 1500 Field Dissolved Oxygen
- FT 1600 Field Turbidity

2. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques:

3. Wells without Plumbing: These wells require that equipment be brought to the well to purge and sample unless dedicated equipment is placed in the well.

~~4. Wells with In-Place Plumbing: Wells with plumbing do not require that equipment be brought to the well to purge and sample. In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply. They are generally found at wellfields, industrial facilities, and private residences. See FS 2300 for procedures to sample potable water wells. Air Strippers or Remedial Systems: These types of systems are installed as remediation devices. Sample these wells like drinking water wells (see FS 2300).~~

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FS 2201 *Equipment and Supplies*

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. In selecting groundwater purging and sampling equipment, give consideration to the depth of the well, the depth to groundwater, the volume of water to be evacuated, the sampling and purging technique, and the analytes of interest. Refer to Tables FS 1000-1, FS 1000-2, FS 1000-3 and FS 2200-1 for selection of appropriate equipment.

Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

1. **FLOW CONTAINER:** DEP recommends using a flow-through cell or container when collecting measurements for purging stabilization. The design must ensure that fresh formation water continuously contacts the measuring devices and does not aerate the sample or otherwise affect the groundwater properties.
2. **PUMPS:** All pumps or pump tubing must be lowered and retrieved from the well slowly and carefully to minimize disturbance to the formation water. This is especially critical at the air/water interface. Avoid the resuspension of sediment particles (turbidity) at the bottom of the well or adhered to the well casing during positioning of the pump or tubing.

2.1 Above-Ground Pumps

2.1.1 Variable Speed Peristaltic Pump: Use a variable speed peristaltic pump to purge groundwater from wells when the static water level in the well is no greater than 20-25 feet below land surface (BLS). If the water levels are deeper than 18-20 feet BLS, the pumping velocity will decrease.

2.1.1.1 A variable speed peristaltic pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.1.1.2 Most analyte groups can be sampled with a peristaltic pump if the tubing and pump configurations are appropriate. See Table FS 1000-3 for proper tubing selection and pump configurations.

2.1.2 Variable Speed Centrifugal Pump: A variable speed centrifugal pump can be used to purge groundwater from 2-inch and larger internal diameter wells. Do not use this type of pump to collect groundwater samples.

2.1.2.1 When purging is complete, do not allow the water that remains in the tubing to fall back into the well. Install a check valve at the end of the purge tubing, and withdraw the tubing slowly from the well while the pump is still running.

2.1.2.2 See Table FS 1000-3 for proper tubing selection and allowable analyte groups.

2.2 Submersible Pumps

2.2.1 Variable Speed Electric Submersible Pump: A variable speed submersible pump can be used to purge and sample groundwater from 2-inch and larger internal diameter wells.

2.2.1.1 A variable speed submersible pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or

formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.1.2 Make sure that the pump housing, fittings, check valves and associated hardware are constructed of stainless steel. Make sure that any other materials are compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.1.3 Install a check valve at the output side of the pump to prevent backflow.

2.2.1.4 If purging and sampling for organics:

- The entire length of the delivery tube must be Teflon, Polyethylene or Polypropylene (PP) tubing.
- The electrical cord must be sealed in Teflon, Polyethylene or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or be constructed of stainless steel.
- All interior components that contact the sample water (impeller, seals, gaskets, etc.) must be constructed of stainless steel or Teflon.

2.2.2 Variable Speed Bladder Pump: A variable speed positive displacement bladder pump (no-gas contact) can be used to purge and sample groundwater from 3/4-inch and larger internal diameter wells.

2.2.2.1 A variable speed bladder pump used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

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2.2.2.2 The bladder pump system is composed of the pump, the compressed air tubing, the water discharge tubing, the controller and a compressor or compressed gas supply.

2.2.2.3 The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder and two (2) check valves. These parts can be composed of various materials, usually combinations of polyvinyl chloride (PVC), Teflon, Polyethylene, PP and stainless steel. Other materials must be compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.2.4 If purging and sampling for organics:

- The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, Polyethylene or PP.
- The entire length of the delivery tube must be Teflon, Polyethylene or PP.
- Any cabling must be sealed in Teflon, Polyethylene or PP, or be constructed of stainless steel.
- Permanently installed pumps may have a PVC pump body as long as the pump remains in contact with the water in the well.

3. BAILERS:

3.1 Purging: DEP does not recommend using bailers for purging unless no other equipment can be used or purging with a bailer has been specifically authorized by a DEP program, permit, contract or order (see Table FS 2200-3). Use a bailer if there is non-aqueous phase liquid (free product) in the well or non-aqueous phase liquid is suspected to

be in the well. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. If a bailer is used, follow FS 2213, section 4, with no deviations.

3.2 Sampling: Bailers may be used to routinely collect some analyte groups or under specific circumstances for other analyte groups (see Table FS 2200-3).

3.3 Construction and Type:

3.3.1 Bailers must be constructed of materials compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

3.3.2 Stainless steel, Teflon, Polyethylene and PP bailers may be used to sample all analytes.

3.3.3 Use disposable bailers when sampling grossly contaminated sample sources.

3.3.4 DEP recommends using dual check valve bailers when collecting samples.

3.3.5 Use bailers with a controlled flow bottom when collecting volatile organic samples.

3.3.6 Use bailers that can be pressurized when collecting filtered samples for metals.

3.4 Contamination Prevention:

3.4.1 Keep the bailer wrapped (foil, butcher paper, etc.) until just before use.

3.4.2 Use protective gloves to handle the bailer once it is removed from its wrapping.

3.4.3 Handle the bailer by the lanyard to minimize contact with the bailer surface.

4. LANYARDS

4.1 Lanyards must be made of non-reactive, non-leachable material such as cotton twine, nylon, or stainless steel; or, coated with Teflon, Polyethylene or PP.

4.1.1 Evaluate the appropriateness of the lanyard material with analyses of equipment blanks for the analytes of interest, as necessary.

4.2 Discard cotton twine, nylon, and non-stainless steel braided lanyards after sampling each monitoring well.

4.3 Decontaminate stainless steel, coated Teflon, Polyethylene and PP lanyards between monitoring wells (see FC 1003). They do not need to be decontaminated between purging and sampling operations.

4.4 Securely fasten lanyards to downhole equipment (bailers, pumps, etc.).

4.5 Do not allow lanyards used for downhole equipment to touch the ground surface.

FS 2210. GROUNDWATER PURGING

Perform procedures in the following sections to calculate purging parameters and to purge groundwater from monitoring wells, wells with installed plumbing, high-volume wells, air stripper systems and other remedial treatment systems.

FS 2211 *Water Level and Purge Volume Determination*

Collect representative groundwater samples from the aquifer. The amount of water that must be purged from a well is determined by the volume of water and/or field parameter stabilization.

1. GENERAL EQUIPMENT CONSIDERATIONS

1.1 Selection of appropriate purging equipment depends on the analytes of interest, the well diameter, transmissivity of the aquifer, the depth to groundwater and other site conditions.

1.2 Use a pump to purge the well.

1.3 Use a bailer if there is non-aqueous phase liquid in the well or non-aqueous phase liquid is suspected to be in the well.

1.4 Bailers may be used if approved by a DEP program, or if bailer use is specified in a permit, contract or DEP order (see Table FS 2200-3). If used, bailers must be of appropriate type and construction, and the user must follow the procedure outlined in FS 2213, section 4, with no deviations. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the DEP program or project manager. DEP does not recommend using bailers for the following reasons:

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1.4.1 Introduces atmospheric oxygen which precipitates metals (i.e., iron) or causes other changes in the chemistry of the water in the sample (i.e., pH)

1.4.2 Agitates groundwater which biases volatile and semi-volatile organic analyses due to volatilization

1.4.3 Agitates the water in the aquifer and resuspends fine particulate matter

1.4.4 Surges the well, loosening particulate matter in the annular space around the well screen

1.4.5 Introduces dirt into the water column if the sides of the casing wall are scraped

2. INITIAL INSPECTION

2.1 Verify the identification of the monitoring well by examining markings, sign plates, placards or other designations.

2.2 Remove the well cover and remove all standing water around the top of the well casing (manhole) before opening the well cap.

2.3 Inspect the exterior protective casing of the monitoring well for damage and document the results of the inspection if there is a problem.

2.4 It is recommended that you place a protective covering around the well head. Replace the covering if it becomes soiled or ripped.

2.5 Inspect the well lock and determine whether the cap fits tightly. Replace the cap if necessary.

3. WATER LEVEL MEASUREMENTS: Use an electronic probe or chalked tape to determine the water level.

3.1 General Procedures

Perform these steps using either the electronic probe or chalked tape method.

3.1.1 Decontaminate all equipment that will contact the groundwater in the well before use.

3.1.2 Measure the depth to groundwater from the top of well casing to the nearest 0.01 foot and always measure from the same reference point or survey mark on the well casing. If there is no reference mark, measure from the north side of the casing.

3.1.3 Record the measurement and the reference point.

3.2 Electronic Probe

3.2.1 Follow the manufacturer's instructions for use.

3.2.2 Record the measurement.

~~3.3 Chalked Line Method: This method is not recommended if collecting samples for organic or inorganic parameters.~~

~~3.3.1 Lower chalked tape into the well until the lower end is in the water (usually determined by the sound of the weight hitting the~~

~~3.3.2 Record the length of the tape relative to the~~ TD
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~~3.3.3 Quickly remove the tape from the well.~~

~~3.3.4 Record the length of the wetted portion to the nearest 0.01 foot.~~

~~3.3.5 Determine the depth to water by subtracting the length of the wetted portion (see section 3.5.3 above) from the total length (see section 3.5.2 above). Record the result.~~

4. WATER COLUMN DETERMINATION

4.1 Do not determine the total depth of the well by lowering the probe to the bottom of the well immediately before purging and sampling. If the well must be sounded, delay purging and sampling activities for at least 24 hours after the well was sounded or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternatively, collect samples before sounding the well.

4.2 Subtract the depth to the top of the water column from the total well depth to determine the length of the water column.

4.3 The total well depth depends on the well construction. Some wells may be drilled in areas of sinkhole or karst formations or rock leaving an open borehole. Attempt to find the total borehole depth in cases where there is an open borehole below the cased portion.

5. WELL WATER VOLUME

5.1 Calculate the total volume of water in gallons in the well using the following equation:

$$V = (0.041)d \times d \times h$$

Where: V = volume in gallons

d = well diameter in inches

h = height of the water column in feet

5.2 The total volume of water in the well may also be determined with the following equation by using a casing volume per foot factor (Gallons per Foot of Water) for the appropriate diameter well:

$$V = [\text{Gallons per Foot of Water}] \times h$$

Where: V = volume in gallons

h = height of the water column in feet

Casing Internal Diameter	Approximate Gallons per Foot of Water
0.75"	0.02
1"	0.04
1.25"	0.06
2"	0.16
3"	0.37
4"	0.65
5"	1.02
6"	1.47
12"	5.88

5.3 Record all measurements and calculations in the field records.

6. Purging Equipment Volume

Calculate the total volume of the pump, associated tubing and container that is used for in situ measurements (flow container), if used, using the following equation:

$$V = p + ((0.041)d \times d \times l) + fc$$

Where: V = volume in gallons
 p = volume of pump in gallons
 d = tubing diameter in inches
 l = length of tubing in feet
 fc = volume of flow cell in gallons

7. When collecting samples from multiple wells on a site, if the groundwater elevation data are to be used to construct groundwater elevation contour maps, all water level measurements must be taken within the same 24-hour time interval unless a shorter time period is required by a DEP program. If the site is tidally influenced, complete the water level measurements within the time frame of an incoming or outgoing tide.

FS 2212 *Well Purging Techniques*

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the water level in the well and minimize the hydraulic stress to the hydrogeologic formation.

Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

A flowchart which summarizes purging procedure options is presented in Figure FS 2200-2.

Select equipment using the construction and configuration requirements specified in Table FS 2200-1. See the discussions in FS 2201.

1. MEASURING THE PURGE VOLUME: The volume of water that is removed during purging must be recorded. Measure the volume during the purging operation.

1.1 Collect the water in a graduated container and multiply the number of times the container was emptied by the volume of the container, or

1.2 Estimate the volume based on pumping rate. Use this technique only if the pumping rate is constant. Determine the pumping rate by measuring the amount of water that is pumped for a fixed period of time or use a flow meter.

1.2.1 Calculate the amount of water that is discharged per minute:

$$D = \frac{\text{Measured amount}}{\text{Total time in minutes}}$$

1.2.2 Calculate the time needed to purge one (1) well volume or one (1) purging equipment volume:

$$\text{Time} = \frac{V}{D}$$

Where: V = well volume determined from FS 2211, section 5, or purging equipment volume

D = discharge rate calculated in section 1.2.1. above

1.2.3 Make new measurements (see section 1.2.1 above) each time the pumping rate is changed, or

1.3 Use a totalizing flow meter.

1.3.1 Record the reading on the totalizer prior to purging.

1.3.2 Record the reading on the totalizer at the end of purging.

1.3.3 Subtract the reading on the totalizer prior to purging from the reading on the totalizer at the end of purging to obtain the volume purged.

1.4 Record in the field records the times that purging begins and ends.

2. Stabilization Measurement Frequency

2.1 Begin to record stabilization measurements after pumping the minimum volume as prescribed in options 2.3 – 2.5 below. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

2.2 If the well screened interval is not known, use option 2.3, below.

2.3 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed at the Top of the Water Column (conventional purge): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Allow at least one quarter (1/4) well volume to purge between subsequent measurements.

2.4 Wells with Fully Submerged Screen and Pump or Intake Tubing Placed Within the Screened Interval (minimizing purge volume): Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) volume of the pump, associated tubing and flow container (if used) prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner

than two (2) minutes apart. Purge at least three (3) volumes of the pump, associated tubing and flow container, if used, prior to collecting a sample.

If the water level drops into the screened interval during purging, lower the pump or tubing intake as in FS 2213, section 1.3 below and follow purging procedures for partially submerged well screens (2.5 below).

2.5 Wells with a Partially Submerged Well Screen: Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner than two (2) minutes apart.

3. PURGING COMPLETION: DEP recommends the use of a flow-through container to measure the stabilization parameters discussed below. Alternatively, measure all parameters *in situ* by inserting measurement probes into the well at the depth appropriate for the purging option. Purging is considered complete if the criteria in section 3.1, 3.2 or 3.3 below are satisfied. Make every attempt to satisfy the criteria in section 3.1. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

3.1 Three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits. The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements of temperature, pH and specific conductance cannot exceed the stated limits. The last three consecutive measurements of dissolved oxygen and turbidity must all be at or below the listed thresholds.

- Temperature: $\pm 0.2^{\circ} \text{C}$
- pH: ± 0.2 Standard Units
- Specific Conductance: $\pm 5.0\%$ of reading
- Dissolved Oxygen: $\leq 20\%$ Saturation
- Turbidity: ≤ 20 NTU

3.2 Naturally occurring conditions may prevent attaining the $\leq 20\%$ saturation criterion for dissolved oxygen, typically in surficial aquifers. See section 3.5, below.

3.3 Naturally occurring conditions may prevent attaining the ≤ 20 NTU criterion for turbidity. However, when collecting groundwater samples for metals or certain inorganic (e.g., phosphorus forms) or extractable organic (e.g. polynuclear aromatic hydrocarbons) chemicals, make every attempt to reduce turbidity to ≤ 20 NTU to avoid a potential turbidity-associated bias for these analytes. See section 3.5, below.

3.4 Document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.

- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5 If the criteria in section 3.1 above for dissolved oxygen and/or turbidity cannot be met, then three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits.

3.5.1 The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements cannot exceed the stated limits.

- Temperature: $\pm 0.2^{\circ} \text{C}$
- pH: ± 0.2 Standard Units
- Specific Conductance: $\pm 5.0\%$ of reading
- Dissolved Oxygen: $\pm 0.2 \text{ mg/L}$ or 10%, whichever is greater
- Turbidity: $\pm 5 \text{ NTUs}$ or 10%, whichever is greater

3.5.2 Additionally, document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of conditions at the site that cause the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5.3 If from review of the submitted data the Department determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the Department cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.6 If the stabilization parameters in either section 3.1 or 3.2 cannot be met, and all attempts have been made to minimize the drawdown, check the instrument condition and calibration, purging flow rate and all tubing connections to determine if they might be affecting the ability to achieve stable measurements. All measurements that were made during the attempt must be documented. The sampling team leader may decide whether or

not to collect a sample or to continue purging after five (5) well volumes (conventional purge section 2.1 or 2.3 above) or five (5) volumes of the screened interval (minimizing purge volumes in section 2.2 above).

Further, the report in which the data are submitted must include the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- Drawdown in the well, if any.
- A description of conditions at the site that caused the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that caused the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

If from review of the submitted data the DEP determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the DEP cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.7 One fully dry purge (not recommended). This criterion applies only if purging was attempted per FS 2212, FS 2213, and section 3.4.1 below, and if it is impossible to balance the pumping rate with the rate of recharge at very low pumping rates (< 100 mL/minute).

3.7.1 If wells have previously and consistently purged dry, when purged according to FS 2212 and FS 2213, and the current depth to groundwater indicates that the well will purge dry during the current sampling event, minimize the amount of water removed from the well by using the same pump to purge and collect the sample:

- 3.7.1.1 Place the pump or tubing intake within the well screened interval.
- 3.7.1.2 Use very small diameter Teflon, Polyethylene or PP tubing and the smallest possible pump chamber volume to minimize the total volume of water pumped from the well and to reduce drawdown.
- 3.7.1.3 Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.
- 3.7.1.4 Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.

- 3.7.1.5 Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).
 - 3.7.1.6 Measure pH, Specific Conductance, Temperature, Dissolved Oxygen and Turbidity and begin to collect the samples (see FS 2222).
4. Collect samples immediately after purging is complete.
- 4.1 The time period between completing the purge and sampling cannot exceed six (6) hours.
 - 4.2 If sample collection does not occur within one (1) hour of purging completion, re-measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity just prior to collecting the sample.
 - 4.2.1 If the measured values are not within 10 percent of the previous measurements, re-purge the well.
 - 4.2.2 See section 3.4 above when collecting samples from wells that have purged dry.

FS 2213 *Purging Wells Without Plumbing (Monitoring Wells)*

1. TUBING/PUMP PLACEMENT

- 1.1 Do not lower the pump or intake hose (tubing) to the bottom of the well. Pump or tubing placement procedures will be determined by the purging option selected in FS 2212, section 2 above or FS 2214 below.
 - 1.1.1 Minimizing Purge Volume: If the following conditions can be met, position the intake hose (tubing) or pump in the screened or open borehole interval.
 - The same pump must be used for both purging and sampling,
 - The well screen or borehole interval must be less than or equal to 10 feet, and
 - The well screen or borehole must be fully submerged.
 - 1.1.2 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 below.
 - 1.1.3 Position the pump or intake hose when purging large-diameter deep wells with open boreholes using the procedure in FS 2214 below.
- 1.2 Conventional Purging: Position the pump or intake tubing in the top one foot of the water column or no deeper than necessary for the type of pump.
 - 1.2.1 If purging with a bailer, see section 4 below.
- 1.3 Partially Submerged Screened Interval: If the well screen or open borehole is partially submerged, and the pump will be used for both purging and sampling, position the pump or intake hose (tubing) in the portion of the water column within the submerged screened or open borehole interval.
 - 1.3.1 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 above.
 - 1.3.2 Purge large-volume, high-recharge wells as in FS 2214 below.
 - 1.3.3 If purging with a bailer, see section 4 below.

2. NON-DEDICATED (PORTABLE) PUMPS

2.1 Variable Speed Peristaltic Pump

- 2.1.1 Install a new, 1-foot maximum length of silicone tubing in the peristaltic pump head.
- 2.1.2 Attach a short section of tubing to the discharge side of the pump-head silicone tubing and into a graduated container.
- 2.1.3 Attach one end of a length of new or precleaned transport tubing to the intake side of the pump head silicone tubing.
- 2.1.4 Place the transport tubing in the monitoring well per one of the options in FS 2213, section 1 above.
- 2.1.5 Measure the depth to groundwater at frequent intervals.
- 2.1.6 Record these measurements.
- 2.1.7 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
- 2.1.8 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- 2.1.9 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.
- 2.1.10 Record the purging rate each time the rate changes.
- 2.1.11 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 2.1.12 Record this measurement.
- 2.1.13 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.2 Variable Speed Centrifugal Pump

- 2.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- 2.2.2 Place the decontaminated suction hose so that water is always pumped from the top of the water column.
- 2.2.3 Equip the suction hose with a foot valve to prevent purge water from re-entering the well.
- 2.2.4 Measure the depth to groundwater at frequent intervals.
- 2.2.5 Record these measurements.
- 2.2.6 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
- 2.2.7 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.
- 2.2.8 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

- 2.2.9 Record the purging rate each time the rate changes.
- 2.2.10 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 2.2.11 Record this measurement.
- 2.2.12 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

~~2.3 Variable Speed Electric Submersible Pump~~

- ~~2.3.1 Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.~~
- ~~2.3.2 Carefully position the decontaminated pump per one of the options in FS 2213, section 1 above.~~
- ~~2.3.3 Measure the depth to groundwater at frequent intervals.~~
- ~~2.3.4 Record these measurements.~~
- ~~2.3.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.~~
- ~~2.3.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.~~
- ~~2.3.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.~~
- ~~2.3.8 Record the purging rate each time the rate changes.~~
- ~~2.3.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.~~
- ~~2.3.10 Record this measurement.~~
- ~~2.3.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.~~

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~~2.4 Variable Speed Bladder Pump~~

- ~~2.4.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.~~
- ~~2.4.2 Attach the tubing and carefully position the pump per one of the options in FS 2213, section 1 above.~~
- ~~2.4.3 Measure the depth to groundwater at frequent intervals.~~
- ~~2.4.4 Record these measurements.~~
- ~~2.4.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.~~
- ~~2.4.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.~~
- ~~2.4.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.~~
- ~~2.4.8 Record the purging rate each time the rate changes.~~

- 2.4.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 2.4.10 Record this measurement.
- 2.4.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.
3. DEDICATED PORTABLE PUMPS: Place dedicated pumps per one of the options in FS 2213, section 1 above.
- 3.1 Variable Speed Electric Submersible Pump
- 3.1.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- 3.1.2 Measure the depth to groundwater at frequent intervals.
- 3.1.3 Record these measurements.
- 3.1.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown. TD
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- 3.1.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.
- 3.1.6 Record the purging rate each time the rate changes.
- 3.1.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 3.1.8 Record this measurement.
- 3.2 Variable Speed Bladder Pump
- 3.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.
- 3.2.2 Measure the depth to groundwater at frequent intervals.
- 3.2.3 Record these measurements.
- 3.2.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.
- 3.2.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.
- 3.2.6 Record the purging rate each time the rate changes.
- 3.2.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 3.2.8 Record this measurement.
4. BAILERS: DEP recommends against using bailers for purging except as a last contingency, or if free product is present in the well or suspected to be in the well. However, they may be used if approved by a DEP program, or specified in a permit, contract or DEP order (see Table FS 2200-3 and FS 2211, section 1.3). If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.
- 4.1 Minimize handling the bailer as much as possible.
- 4.1.1 Remove the bailer from its protective wrapping just before use.
- 4.1.2 Attach a lanyard of appropriate material (see FS 2201, section 4).

- 4.1.3 Use the lanyard to move and position the bailer.
- 4.2 Lower and retrieve the bailer slowly and smoothly.
- 4.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column.
- 4.3.1 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column. Ensure that the length of the bailer does not exceed the length of the water column.
- 4.3.2 Allow time for the bailer to fill with water as it descends into the water column.
- 4.4 Carefully raise the bailer.
- 4.4.1 Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.
- 4.5 Measure the purge volume by one of the methods outlined in FS 2212, section 1.
- 4.5.1 Record the volume of the bailer.
- 4.6 Continue to carefully lower and retrieve the bailer as described above until the purging completion conditions specified in FS 2212, section 3, have been satisfied.
- 4.6.1 Remove at least one (1) well volume before collecting measurements of the field parameters. Take each subsequent set of measurements after removing at least one quarter (1/4) well volume between measurements.

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FS 2214 *Purging Large-Volume, High-Recharge Wells With Portable Pumps*

If a well originally constructed for high-flow-rate pumping will be sampled as a monitoring well, use these guidelines to develop a purging procedure applicable to the specific details of the well construction. Typical wells constructed for this purpose may be deep, large-diameter wells with a section of open borehole. Evaluate each well on a case-by-case basis and consider any available information on the construction and hydraulic performance of the well.

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1. PURGING PROCEDURE

- 1.1 Place the pump at the top of the open borehole segment of the well.
- 1.2 Start purging while monitoring stabilization parameters as in FS 2212, section 3 above.
- 1.3 Purge at least one equipment volume before measuring stabilization parameters.
- 1.4 If the well is being purged for the first time using these guidelines, monitor stabilization parameters for an extended period until confident that sufficient volume has been pumped from the open borehole to draw fresh formation water into the pump tubing and flow-through container. Use the information obtained from the first-time purging of the well to determine the pumping rate and duration of purging required for future sampling events at the well.
- 1.5 Purge at least three equipment volumes before evaluating purging completion.

2. PURGING COMPLETION

2.1 Complete the purging of the well when the last three consecutive measurements of the purge stabilization parameters have met the applicable criteria specified in FS 2212, section 3 above.

3. Collect samples from the well using the procedures in FS 2221, section 1 below.

FS 2215. *Purging Wells With Plumbing (production wells or permanently installed pumps equipped with sampling ports or sampling spigots)*

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible. When purging is required and the purge volume of the plumbing system is not known, purge the system until the purging completion criteria in FS 2212, section 3, have been met.

1. CONTINUOUSLY RUNNING PUMPS

1.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

1.2 Remove all hoses, aerators TD
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1.3 Open the spigot and purge at maximum flow.

1.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

1.5 If the spigot is before any storage tank, purge until sufficient volume is removed to flush the stagnant water from the spigot and the tap line to the spigot.

1.6 Reduce the flow rate to ≤ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to ≤ 100 mL/minute before collecting the samples.

2. INTERMITTENTLY RUNNING PUMPS

2.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

2.2 Remove all hoses, aerators and filters (if possible).

2.3 Open the spigot and purge sufficient volume at a maximum, practical flow rate to flush the spigot and lines and until the purging completion criteria in FS 2212, section 3, have been met.

2.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

2.5 Ensure that the purge stabilization measurement of dissolved oxygen is not biased with aeration of the sample by a high flow rate in the flow-through container.

2.6 Reduce the flow rate to ≤ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to ≤ 100 mL/minute before collecting the samples.

FS 2216. *Purging Airstrippers and Remedial Treatment Systems*

If collecting samples for groundwater contamination monitoring, follow FS 2215 above.

FS 2220. GROUNDWATER SAMPLING TECHNIQUES

1. Purge wells using the techniques outlined in FS 2210.
2. Replace the protective covering around the well if it is soiled or torn after completing the purging operations.
3. EQUIPMENT CONSIDERATIONS

Follow all notes and restrictions as indicated in Table FS 2200-1 and as discussed in FS 2201.

NOTE: The only pumps that are currently approved for use in collecting volatile organic samples through the pump are stainless steel and Teflon variable speed submersible pumps, stainless steel and Teflon or Polyethylene variable speed bladder pumps, and permanently installed PVC bodied pumps (variable speed bladder or submersible pumps) as long as the pump remains in contact with the water in the well at all times.

- 3.1 Collect the sample into the sample container from the sampling device. **Do not** use intermediate containers.
- 3.2 In order to avoid contaminating the sample or loss of analytes from the sample:
- 3.3 Handle the sampling equipment as little as possible.
 - 3.3.1 Minimize the equipment that is exposed to the sample.
 - 3.3.2 Minimize aeration of samples collected for VOC analysis.
 - 3.3.3 Reduce sampling pump flow rates to ≤ 100 mL/minute when collecting VOC samples.
- 3.4 Dedicated Sampling Equipment
 - 3.4.1 Whenever possible, use dedicated equipment because it significantly reduces the chance of cross-contamination.
 - 3.4.2 Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).
 - 3.4.3 All material construction and restrictions from Table FS 2200-1 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.
- 3.5 Cleaning/Decontamination
 - 3.5.1 Clean or ensure dedicated pumps are clean before installation. They do not need to be cleaned prior to each use but must be cleaned if they are withdrawn for repair or servicing.
 - 3.5.2 Clean or make sure any permanently mounted tubing is clean before installation.
 - 3.5.3 Change or clean tubing when the pump is withdrawn for servicing.
 - 3.5.4 Clean any replaceable or temporary parts as specified in FC 1000.
 - 3.5.5 Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.
 - 3.5.6 Clean or ensure dedicated bailers are clean before placing them into the well.
 - 3.5.7 Collect an equipment blank on dedicated bailers before introducing them into the water column.

3.5.8 Suspend dedicated bailers above the water column if they are stored in the well.

FS 2221. *Sampling Wells Without Plumbing*

1. SAMPLING WITH PUMPS: Variable speed stainless steel and Teflon submersible pumps and stainless steel, Teflon or Polyethylene bladder pumps, and permanently installed PVC-bodied variable speed submersible or bladder pumps, as long as the pump remains in contact with the water in the well at all times, may be used to sample for all organics. The delivery tubing must be Teflon, Polyethylene or PP. **Extractable organics** may be collected through a peristaltic pump if ≤ 1 foot of silicone tubing is used in the pump head or a vacuum trap is used (see Figure FS 2200-1 for specific configuration). Follow all notes and restrictions as defined in Table FS 2200-1 and discussed in Equipment and Supplies (FS 2201) when using pumps to collect samples.

Do not lower the pump or tubing to the bottom of the well.

1.1 Peristaltic Pump

1.1.1 Volatile Organics Using Manual Fill and Drain Method: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.1.3 through 1.1.1.6 below are prohibited.

- 1.1.1.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.
- 1.1.1.2 Remove the drop tubing from the inlet side of the pump.
- 1.1.1.3 Submerge the drop tubing into the water column and allow it fill.
- 1.1.1.4 Remove the drop tubing from the well.
- 1.1.1.5 Prevent the water in the tubing from flowing back into the well.
- 1.1.1.6 Carefully allow the groundwater to drain by gravity into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.
- 1.1.1.7 Repeat steps 1.1.1.3 - 1.1.1.6 until enough vials are filled.

1.1.2 Volatile Organics Using the Pump to Fill and Drain the Tubing: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.2.2 through 1.1.2.8 below are prohibited.

- 1.1.2.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.
- 1.1.2.2 Submerge the drop tubing into the water column.
- 1.1.2.3 Use the pump to fill the drop tubing.
- 1.1.2.4 Quickly remove the tubing from the pump.
- 1.1.2.5 Prevent the water in the tubing from flowing back into the well.
- 1.1.2.6 Remove the drop tubing from the well and fill the vials using the pump or gravity-drain methods in steps 1.1.2.7 or 1.1.2.8 below.
- 1.1.2.7 Reverse the flow on the peristaltic pump to deliver the sample into the vials at a slow, steady rate. The flow rate must be ≤ 100 mL/minute.

1.1.2.8 Or, remove the drop tubing from the inlet side of the pump and carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be ≤ 100 mL/minute.

1.1.2.9 Repeat steps 1.1.2.2 through 1.1.2.8 until enough vials are filled.

1.1.3 Extractable Organics Collected Through Silicone Pump-Head Tubing:

1.1.3.1 Ensure that a 1-foot maximum length of new silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.1.3.2 Collect extractable organic samples directly from the effluent delivery tubing (attached to discharge side of the silicone pump head tubing) into the sample container.

1.1.3.3 If there is a concern that sample analytes are absorbed, adsorbed, leached or otherwise affected or lost by pumping through the silicone pump-head tubing, sample the well using the organic trap assembly in 1.1.4 below.

1.1.4 Extractable Organics Using an Optional Organic Trap Assembly

1.1.4.1 Assemble the components of the pump and trap according to Figure FS 2200-1.

1.1.4.2 The sample container should be the trap bottle.

1.1.4.3 All equipment that contacts the groundwater **before** the sample container must be constructed of Teflon, Polyethylene, PP, stainless steel or glass, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. **Do not use a rubber stopper as a cap.**

1.1.4.4 Connect the outflow tubing from the container to the influent side of the peristaltic pump.

1.1.4.5 Prevent the water in the down-hole delivery tubing from flowing back into the well while performing this connection.

1.1.4.6 Turn the pump on and reduce the flow rate to a smooth and even flow.

1.1.4.7 Discard a small portion of the sample to allow an air space.

1.1.4.8 Preserve (if required), label and complete the field notes.

1.1.5 Inorganics

1.1.5.1 Inorganic samples may be collected from the effluent tubing.

1.1.5.2 If samples are collected from the pump, decontaminate all tubing (including the tubing in the head) or change it between wells.

1.1.5.3 Preserve (if required), label and complete field notes.

1.2 Variable Speed Bladder Pump

1.2.1 If sampling for organics the pump body must be constructed of stainless steel and the valves and bladder must be Teflon. All tubing must be Teflon, Polyethylene, or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or made of stainless steel.

1.2.2 After purging to a smooth even flow, reduce the flow rate.

1.2.3 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

1.3 Variable Speed Submersible Pump

1.3.1 The housing must be stainless steel.

1.3.2 If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon, Polyethylene or PP. The delivery tubing must be Teflon, Polyethylene or PP and the electrical cord must be sealed in Teflon and any cabling must be sealed in Teflon or constructed of stainless steel.

1.3.3 After purging to a smooth even flow, reduce the flow rate.

1.3.4 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

2. SAMPLING WITH BAILERS: A high degree of skill and coordination are necessary to collect representative samples with a bailer. When properly used, bailers may be used to collect samples for certain analyte groups and under specific conditions (see Table FS 2200-3). They must be of an appropriate type and construction (see FS 2201, section 3), and must be used as outlined below. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

2.1 General Considerations

2.1.1 Minimize handling the bailer as much as possible.

2.1.1.1 Wear sampling gloves.

2.1.1.2 Remove the bailer from its protective wrapping just before use.

2.1.1.3 Attach a lanyard of appropriate material (see FS 2201, section 4).

2.1.1.4 Use the lanyard to move and position the bailers.

2.1.2 Do not allow the bailer or lanyard to touch the ground.

2.1.3 Rinsing

2.1.3.1 If the bailer is certified precleaned, no rinsing is necessary.

2.1.3.2 If both a pump and a bailer are to be used to collect samples, rinse the exterior and interior of the bailer with sample water from the pump before removing the pump.

2.1.3.3 If the purge pump is not appropriate for collecting samples (e.g., non-inert components), rinse the bailer with by collecting a single bailer of the groundwater to be sampled. Use the technique described in section 2.2, Bailing Technique, below.

2.1.3.4 Discard the water appropriately.

2.1.3.5 **Do not** rinse the bailer if Oil & Grease, TRPHs, etc., (see FS 2006) are to be collected.

2.2 Bailing Technique

2.2.1 Collect all samples that are required to be collected with a pump before collecting samples with the bailer.

2.2.2 Raise and lower the bailer gently to minimize stirring up particulate matter in the well and the water column which can increase sample turbidity.

2.2.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column. Ensure that the length of the bailer does not exceed the length of the water column.

2.2.3.1 When the bailer is in position, lower the bailer into the water column at a rate of 2 cm/sec until the desired depth is reached (see section 2.2.3 above).

2.2.4 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column.

2.2.5 Allow time for the bailer to fill with aquifer water as it descends into the water column.

2.2.6 Do not allow the bailer to touch the TD
7/20/11 the well or particulate matter will be incorporated into the sample.

2.2.6.1 Carefully raise the bailer (see section 2.2.2 above). Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

2.2.7 Lower the bailer to approximately the same depth each time.

2.2.8 Collect the sample.

2.2.8.1 Install a device to control the flow from the bottom of the bailer and discard the first few inches of water. Reduce the flow to ≤ 100 mL/minute when collecting VOC samples.

2.2.8.2 Fill the appropriate sample containers by allowing the sample to slowly flow down the side of the container. Minimize aeration of VOC samples.

2.2.8.3 Discard the last few inches of water in the bailer.

2.2.9 Repeat steps 2.2.1 through 2.2.8.3 for additional samples.

2.2.10 Measure the DO, pH, temperature, turbidity and specific conductance after the final sample has been collected.

2.2.10.1 Record all measurements and note the time that sampling was completed.

3. SAMPLING WELLS WITH FLOATING NON-AQUEOUS PHASE LIQUID: DEP does not recommend the sampling of wells with floating non-aqueous phase liquid for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g., solvent) that floats on the water table. Sampling is acceptable if the information is to be used for the purpose of remedial design.

Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, these wells typically do not provide legitimate data because of permanent chemical contamination from product contact with the well casing for an extended period of time.

DEP does reserve the right to require sampling of these wells, not for levels of trace contaminants, but for confirmation of an appropriate remediation technique. This type of sampling is performed **below** the non-aqueous phase layer (see section 3.2 below).

3.1 Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.

3.1.1 Non-aqueous phase liquid is normally sampled for two reasons:

- Documentation for its existence and thickness; and
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product.

3.1.2 Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable Polyethylene and PP bailers are also acceptable. Other wide mouth vessels may be used for sampling non-aqueous phase liquid in an excavation.

3.1.3 Monitoring Well

3.1.3.1 If a non-aqueous phase liquid is identified in a monitoring well during the water level measurement, measure its thickness in the well. If the thickness of the non-aqueous phase liquid is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer.

3.1.3.2 Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.

3.1.3.3 Pour a portion of the product into a glass sample container.

3.1.3.4 This sample is considered a concentrated waste. Therefore, package the container in protective wrapping to prevent breakage, isolate from other samples, and ice to 4°C.

3.1.4 Excavation

3.1.4.1 If non-aqueous phase liquid is observed in an open excavation, a glass sample container or a precleaned intermediate vessel may be used to collect the sample.

3.1.4.2 Securely tie a lanyard to the container and lower it into the excavation.

3.1.4.3 Gently lower and retrieve the container so that no solid material is released or collected.

3.1.4.4 If sufficient water is available, a bailer can be used.

3.1.4.5 Although not recommended, screened casing can be placed (or augered and placed) in the bottom of the excavation and the product sampled with a bailer.

3.1.4.6 Avoid dangerous situations, such as standing too close to the edge of an excavation, riding in the backhoe bucket, or entering a trench or excavation that may collapse.

3.1.4.7 Follow all applicable OSHA regulations.

3.2 Sampling Below Product

3.2.1 This type of depth-specific sampling to attempt to sample the dissolved constituents in the water column below the product layer is performed only at the request of DEP or its designee.

3.2.2 These data provide information that helps define adequate groundwater treatment. Without these data, incorrect (and sometimes unnecessarily expensive) remediation techniques may be designed for a situation where they are not required.

3.2.3 There are some substantial logistical problems involved with sending a sampler through non-aqueous phase liquid to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of "self-engineered" equipment or coverings may be the best option.

3.2.4 These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.

3.2.5 Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.

3.2.6 Although not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.

3.2.6.1 Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.

3.2.6.2 Introduce the wrapped hose slowly to not entrain any more product into the dissolved layer located below.

3.2.6.3 Also, perform this procedure with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet, realizing that actual sampling may be occurring several feet below the product layer.

FS 2222. *Sampling Low Permeability Aquifers or Wells That Have Purged Dry*

1. Collect the sample(s) after the well has been purged according to FS 2212, section 3.4. Minimize the amount of water removed from the well by using the same pump to purge and collect the sample. If the well has purged dry, collect samples as soon as sufficient sample water is available.
2. Measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity at the time of sample collection.
3. Advise the analytical laboratory and the client that the usual amount of sample for analysis may not be available.

~~**FS 2223.** *Sampling Wells With In-Place Plumbing*~~

- ~~1. If a storage tank is present, locate a cold water spigot, valve or other sampling point close to the well head between the pump and the storage tank. If there is no sampling location between the pump and the storage tank, locate a valve or other sampling point closest to the tank.~~

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- ~~1.1 Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible.~~
- ~~2. Remove all screens or aerators and reduce the flow rate to no more than 500 mL/minute. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less. Collect the samples directly into the appropriate containers.~~

FS 2224. *Sampling Airstripper and Remedial Treatment System Sampling*

1. Reduce the flow rate to less than 500 mL/minute and begin sample collection.
2. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less.
3. Collect the samples directly into the appropriate containers.

FS 2225. *Filtering Groundwater Samples*

Filtered groundwater samples can only be collected after approval from the DEP program or project manager. If filtering is approved, the DEP program or permit condition may require both filtered and unfiltered samples to be collected, analyzed and reported.

1. FILTERING GROUNDWATER FOR METALS:

1.1 Unless specified otherwise by the DEP program, use a new, disposable, high capacity, 1- μ m in-line filter.

1.2 Use a variable speed peristaltic pump or submersible pump with the in-line filter fitted on the outlet end.

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1.2.1 Peristaltic pumps, bladder pumps or submersible pumps can be used when water levels are no greater than 20 to 25 feet deep.

1.2.2 Bladder pumps or submersible pumps must be used when water levels are greater than 20 to 25 feet deep.

1.3 Ensure that a 1-foot maximum length of new, silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.4 Ensure that new or precleaned delivery tubing was assembled with the peristaltic pump before the well was purged if the same pump is being used to purge and sample the well. Otherwise, assemble the pump with new or precleaned delivery tubing and the new filter.

1.5 Insert the filter on the high pressure side (i.e., on the delivery side) of the pump.

1.5.1 Flush the filter before attaching to the pump tubing assembly with 30-50 mL of analyte free water or an inert gas (nitrogen) to remove atmospheric oxygen;

1.5.2 Or, with the filter attached to the pump tubing assembly, hold the filter upright with the inlet and outlet in the vertical position and pump water from the aquifer through the filter until all atmospheric oxygen has been removed.

1.6 Collect the filtered samples directly into the sample container from the high-pressure (delivery) side of the pump tubing assembly.

1.6.1 Collect filtered samples by either of the methods in 1.6.1.3 or 1.6.1.4 below if the static water level in the well is too deep for a variable speed peristaltic pump and a variable speed electric submersible pump or variable speed bladder pump is not available.

1.6.1.1 Do not agitate the sample or expose it to atmospheric oxygen.

1.6.1.2 **Do not** pour the sample into any intermediate vessel for subsequent filtration.

1.6.1.3 Collect the sample in a Polyethylene, Teflon or PP bailer that can be pressurized. When the bailer has been retrieved, immediately connect the filter and begin to pressurize the bailer;

1.6.1.4 Or, collect the sample with a bailer and immediately place the intake tube of the peristaltic pump into the full bailer and begin pumping the water through the filter as described in section 1.2 above.

1.7 **Do not** use the following equipment for filtering groundwater samples for metals:

1.7.1 Any pump and apparatus combination in which the filter is on the vacuum (suction) side of the pump.

1.7.2 Any type of syringe or barrel apparatus.

1.7.3 Any filter that is not encased in a one-piece, molded unit.

2. Filtering groundwater for non-metallic analytes

2.1 The following analytes cannot be filtered:

- Oil and Grease
- Total Recoverable Petroleum Hydrocarbons (TRPH)
- FL-PRO
- Volatile Organic Compounds (VOC)
- Microbiological Analytes
- Volatile Inorganic Compounds (e.g., Hydrogen Sulfide)

2.2 Unless specified otherwise by the regulatory program, use a new, disposable, high capacity, 0.45 µm in-line filter.

2.3 Assemble the pump, tubing and filter as in 1.2 – 1.5 above.

2.4 Flush the filter as in 1.5.1 or 1.5.2 above.

2.5 Collect the samples as in 1.6 – 1.6.1.4 above.

Appendix FS 2200
Tables, Figures and Forms

Table FS 2200-1 Equipment for Collecting Groundwater Samples

Table FS 2200-2 Dissolved Oxygen Saturation

Table FS 2200-3 Allowable Uses for Bailers

Figure FS 2200-1 Pump and Trap for Extractable Organics

Figure FS 2200-2 Groundwater Purging Procedure

Form FD 9000-24 Groundwater Sampling Log

Table FS 2200-1
Equipment for Collecting Groundwater Samples

Activity	Equipment Type
Well Purging	Variable speed centrifugal pump Variable speed submersible pump Variable speed bladder pump Variable speed peristaltic pump Bailer with lanyard: Not Recommended
Well Stabilization	pH meter DO meter Conductivity meter Thermometer/Thermistor Turbidimeter Flow-through cell Multi-function meters
Sample Collection	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Bailer with lanyard (See Table FS 2200-3)
Filtration	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Pressurized bailer 1.0 µm high capacity molded filter 0.45 µm high capacity molded filter
Groundwater Level	Electronic sensor Chalked tape

Table FS 2200-2
Dissolved Oxygen Saturation

TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L
deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

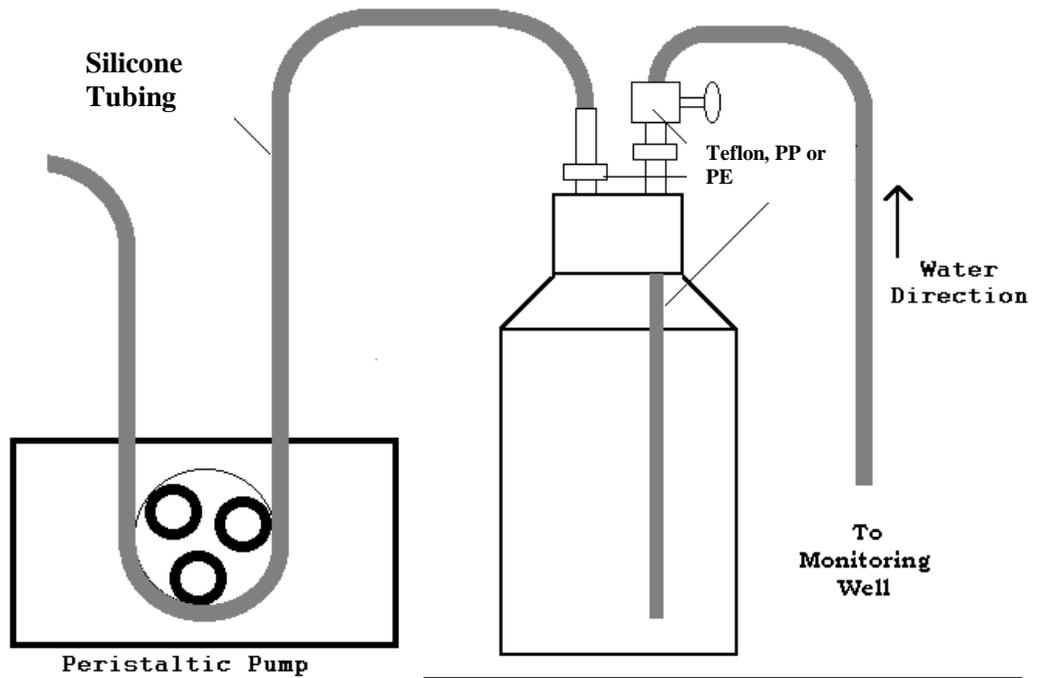
**Table FS 2200-3
 Allowable Uses for Bailers**

• ANALYTE GROUP(S)	• PURGING (Not Recommended)	• SAMPLING	
	Use:	Use:	Not Recommended:
Volatile Organics Extractable Organics Radionuclides, including Radon Metals Volatile Sulfides	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If concentrations exceed action levels, the purpose is to monitor effective treatment, and the DEP program allows the use of bailers; or If specified by DEP permit, program, contract or order. or If operated by a skilled individual with documented training in proper techniques and using appropriate equipment. Field documentation must demonstrate that the procedure in FS 2221, section 2 was followed without deviation.	If concentrations are near or below the stated action levels; or If a critical decision (e.g., clean closure) will be made based on the data; or If data are to demonstrate compliance with a permit or order.
Petroleum Hydrocarbons (TRPH) & Oil & Grease	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	Only if allowed by permit, program, contract or order as samples should be collected into the container without intermediate devices.	Unless allowed by permit, program, contract or order.

DEP-SOP-001/01
FS 2200 Groundwater Sampling

• ANALYTE GROUP(S)	• PURGING (Not Recommended)	• SAMPLING	
	Use:	Use:	Not Recommended:
Biologicals Inorganic Non-Metallics Aggregate Organics Microbiological Physical and Aggregate Properties	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If all analytes collected from the well can be collected with a bailer; or If collected <u>after</u> collecting all analytes that require the use of a pump.	Before collecting any analytes that must be collected with a pump.
Ultra-Trace Metals	Never	Never	

Figure 2200-1
Pump and Trap for Extractable Organics



The glass sample bottle must be threaded to use a reusable sampling cap lined and installed with fittings made of Teflon, polypropylene or polyethylene, similar to the design shown.

DEP-SOP-001/01
FS 2200 Groundwater Sampling

Scenario 1: WELL SCREEN COMPLETELY SUBMERGED

Scenario 2: WELL SCREEN PARTIALLY SUBMERGED

Option 1a: Minimal Purge Volume: Pump or tubing is placed within the middle of the screen interval. The following conditions must be met to use this option:

1. The well screen interval is ≤ 10 feet.
2. Although drawdown may occur in the well when purging is initiated, the drawdown has to stabilize (Aquifer Recovery Rate = Purge Rate).
3. The samples will be obtained with the same equipment that was used to purge the well. Therefore, centrifugal pumps and bailers are not suitable for use in Option 1a.

If one or more of these conditions do not apply, use Option 1b.

Option 1b: Conventional Purge: Pump, tubing, or bailer¹ is placed above the screen at the top of the water column.

¹ DEP does not recommend the use of a bailer for purging; however, if a bailer is used it shall be lowered and raised at the rate of 2 cm/sec in the top of the water column.

Option 2a: A bailer¹ is placed at the top of the water column and is used to purge and sample the well.

Option 2b: Pump or tubing is placed within the middle of the saturated portion of the screen interval.

If the pump or tubing that was used for purging will not be used to obtain the sample, then position the pump or tubing at the top of the water column for purging.

Purging Procedure #1

1. After the drawdown in the well stabilizes, purge at least one equipment volume then collect the first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.
3. Purge at least three equipment volumes before sampling.

Purging Procedure #2

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters \geq every 1/4 well volume.

Purging Procedure #3

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters ≥ 2 to 3 minutes apart.

Purging Completion

If Dissolved Oxygen is $\leq 20\%$ of saturation for the measured temperature and Turbidity is ≤ 20 NTUs, then purging is complete when **three** consecutive readings of the parameters listed below are within the following ranges:

Temperature $\pm 0.2^\circ\text{C}$
pH ± 0.2 Standard Units
Specific Conductance $\pm 5.0\%$ of reading

If Dissolved Oxygen (DO) is $> 20\%$ of saturation for the measured temperature and/or Turbidity is > 20 NTUs after every attempt has been made to reduce DO and/or turbidity, then purging is complete when **three** consecutive readings of the parameters listed below are within the following ranges:

Temperature $\pm 0.2^\circ\text{C}$
pH ± 0.2 Standard Units
Specific Conductance $\pm 5.0\%$ of reading
Dissolved Oxygen ± 0.2 mg/L or readings are within 10% (whichever is greater).
Turbidity ± 5 NTUs or readings are within 10% (whichever is greater).

If the well is expected to purge dry, position the pump or tubing within the screened interval and purge at ≤ 100 mL/minute until two equipment volumes are removed. Use the same pump for purging and sampling.

If the well purges dry at the lowest achievable flow rate (pumping at 100 mL/minute or less), then after a sufficient amount of water recharges in the well, collect the samples.

In either case listed above, before samples are collected, measure (once) pH, temperature, specific conductance, dissolved oxygen, and turbidity.

If one or more parameters do not stabilize after 5 volumes of the screened interval (purging procedure #1) or 5 well volumes (purging procedure #s 2 & 3) are removed, purging may be discontinued at the discretion of the sampling team leader.

FS 3000. SOIL

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FT 1000 – FT 2000 Field Testing and Calibration

1. Introduction and Scope

1.1. Use these SOPs during field investigations to collect soil samples that are representative of current site conditions. It is very important to ensure that the collected samples are neither altered nor contaminated by sampling and handling techniques.

1.2. The following topics include: equipment choice, equipment construction materials, grab and areal or depth composite sampling techniques. Sample collection methods fall into three general depth classifications: surface, shallow subsurface, and deep subsurface. Once the samples are acquired, the handling procedures are very similar and are described below.

2. GENERAL

2.1. Select sampling equipment based on the type of sample to be collected and the analytes of interest. Choose soil sampling locations such that a representative portion of the soil is collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.

2.2. If background and/or quality control sampling is warranted and feasible as determined in the site's work plan or by the project manager, select an up gradient, undisturbed location for obtaining the background and/or quality control samples. Be aware that differences in soil types may affect these background samples (e.g., sands vs. clays).

2.3. **Do not collect** samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, except for waste characterization purposes for disposal.

2.4. Do not use samples that are collected for geological/lithological or vapor meter determinations for chemical analyses.

3. EQUIPMENT AND SUPPLIES

3.1. All equipment must be constructed of materials consistent with the analytes of interest. Refer to FS 1000, Tables FS 1000-1, FS 1000-2 and FS 1000-3 for selection of appropriate equipment and materials.

3.2. For information on sample container size and construction, see FS 1000, Table FS 1000-6.

3.3. For information on sampling equipment cleaning requirements, see FC 1000.

3.4. For information on preservation and holding time requirements, see FS 1000, Table FS 1000-6.

3.5. For information on documentation requirements, see FD 1000.

4. PROCEDURES FOR COMPOSITING

4.1. The following is not a complete discussion regarding all available sampling protocols nor the appropriateness or inappropriateness of compositing soil samples. The appropriateness of compositing soil samples will depend on the data quality objectives of the project. However, it is sometimes advantageous to composite soil samples to minimize the number of samples to be analyzed when sampling highly contaminated areas. Obtain permission from the DEP program.

4.1.1. Select sampling points from which to collect each aliquot.

4.1.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

4.1.3. **Combine the aliquots of the sample directly in the sample container with no pre-mixing.**

4.1.4. Record the amount of each aliquot (volume or weight).

4.1.5. Label container, preserve on wet ice to 4°C and complete field notes.

4.1.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

5. SPECIFIC PROCEDURES FOR VOLATILE ORGANIC COMPOUNDS

Follow the procedures specified in EPA Method 5035 for sample collection and sample preparation. The protocols listed below **do not replace Method 5035** but clarify and/or modify certain method procedures. Therefore, it is essential that all organizations have a copy of Method 5035 as a reference document.

5.1. Container Preparation

5.1.1. All containers must be cleaned according to the FC 1000 sample container cleaning procedures for volatile organics.

5.1.2. Sample Vials: If sample vials are filled in the field, they must be provided with all reagents, stirring devices, label **and vial cap** to be used during sample analysis. These vials must be preweighed by the laboratory and records must be maintained so that there is an unambiguous link between the tare weight and the filled sample vial.

5.2. Collection Procedure

5.2.1. The sample vials (when used) will contain a premeasured amount of liquid. The laboratory must weigh the vials before sending into the field, and must weigh them again after receipt. Therefore:

- Do not lose any of the liquid either through evaporation or spillage
- Do not use a vial if some of the contents has spilled, or if it appears that some has leaked during transport
- Use the laboratory-supplied container label for identification information. **DO NOT apply any additional labels to the container**

- Do not interchange vial caps or septa
- 5.2.2. Minimize exposure to air by obtaining the sample directly from the sample source, using a coring device or a commercially designed sampling tool.
- 5.2.2.1. The sample collection device must be designed to fit tightly against the mouth of the vial or be small enough to be inserted into the vial. Use:
- EnCore or equivalent sampling devices or
 - Disposable plastic syringes with the syringe end cut off prior to sampling (use **once** per sampling location).
- 5.2.2.2. Extrude the sample directly into the sample container.
- 5.2.3. Follow the method procedures for field transfer into the vial.
- 5.2.4. Procedures for determining the sample weight in the field are not required unless the project manager requires an accurate determination of the 5-gram sample size.
- 5.2.4.1. If the vials are returned to the laboratory for weighing, the sampler must be proficient in estimating the requisite 5-gram weight necessary for each sample.
- 5.2.4.2. If an accurate estimate of the 5-gram sample size is desired prior to starting sample collection activities, use a balance with a sensitivity of 0.1 gram. Check the balance calibration before each day's use with a set of weights that have been calibrated against NIST-traceable weights at least annually.
- 5.2.5. If the sampling device is transported to the laboratory with a sample, make sure the seals are intact, especially if collecting samples from sandy soils.
- 5.2.6. Collect at least two replicate samples from the same soil stratum and within close proximity to the original sample location.
- 5.2.7. Collect an additional aliquot of sample for screening and dry weight determinations.
- 5.3. Preservation (see FS 1000, Table FS 1000-7)
- 5.3.1. Low Level ($\leq 200 \mu\text{g}/\text{kg}$ volatile organics)
- 5.3.1.1. Method 5035 discusses the use of sodium bisulfate, which is an acid. Since Florida soils contain significant amounts of calcium carbonate that reacts with acids, DEP does not recommend using this preservative.
- 5.3.1.2. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.
- 5.3.1.3. Analyze unpreserved samples (no acid) within 48 hours.
- 5.3.1.4. Analyze acid-preserved samples within the specified 14-day holding time.
- 5.3.1.5. Analyze unpreserved samples that have been collected in a septum vial with premeasured analyte-free water within 48 hours.
- 5.3.1.6. If unpreserved samples collected in a septum vial with premeasured analyte-free water are frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.
- 5.3.1.7. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.1.8. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2. High Level (> 200 µg/kg volatile organics)

5.3.2.1. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.2.2. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.2.3. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and stored at 4°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2.4. Analyze samples that that have been preserved in methanol in the field within 14-days.

6. BULK SAMPLES: The collection of bulk samples will depend on the data quality objectives of the project.

6.1. Do not composite or mix VOC samples unless required by the DEP program or if mandated by a formal DEP document (permit, order or contract).

6.2. Select sampling points from which to collect each aliquot.

6.3. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

6.3.1. **Combine the aliquots of the sample directly in the sample container with no pre-mixing..**

6.3.2. Pack soil tightly minimizing as much headspace as possible in the sample container.

6.3.3. Cap container tightly with Teflon side facing sample.

6.4. Record the amount of each aliquot (volume or weight) in the field notes.

6.5. Label container. Refer to FS 1000, Table FS 1000-7 for preservation and holding time requirements.

6.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

FS 3100. Surface Soil Sampling

Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.

1. Remove leaves, grass and surface debris from the area to be sampled.
2. Collect samples for volatile organic analyses as described in FS 3000, section 5.
3. Select an appropriate precleaned sampling device and collect the sample.
4. Transfer the sample to the appropriate sample container.
5. Clean the outside of the sample container to remove excess soil.

6. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3200. Subsurface Soil Sampling

Interval begins at approximately 12 inches below ground surface.

FS 3210. SAMPLE COLLECTION PROCEDURE

Use the following after the desired depth has been reached by one of the methods outlined in FS 3220.

1. Collect samples for volatile organic analyses as described in FS 3000, section 5.
2. For other analyses, select an appropriate precleaned sampling device and collect the sample.
3. Transfer the sample to the appropriate sample container.
4. Clean the outside of the sample container to remove excess soil.
5. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3220. REACHING THE APPROPRIATE DEPTH

1. SHOVELS AND DIGGERS: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.
 - 1.1. Dig a hole or trench to the required depth.
 - 1.2. Follow the sample collection procedures outlined in FS 3210.
2. BACKHOE: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.
 - 2.1. Dig a trench to the appropriate depth.
 - 2.2. Expose the sample, in the trench, by using a precleaned spoon, spatula or equivalent to clean away the soil that came in contact with the backhoe bucket.
 - 2.3. Use a **second** precleaned utensil to actually collect the sample from the trench.
 - 2.4. Follow the procedures outlined in FS 3210 to collect the sample.
3. BUCKET AUGERS AND HOLLOW CORERS: Suitable to reach soils from approximately 12 inches to a point when using the implement becomes impractical.
 - 3.1. Push and rotate the auger into the soil until the bucket is filled.
 - 3.2. Addition of a non-contaminating sleeve may allow an undisturbed soil sample to be obtained.
 - 3.2.1. The device consists of a standard auger head with a removable sleeve, which is inserted into the auger barrel. In this case it is the sleeve, which fills with soil.
 - 3.2.2. Remove the sleeve from the auger and cap.
 - 3.3. If the auger hole is prone to collapse due to low cohesion in some soils, DEP recommends inserting a temporary rigid PVC casing into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.

- 3.4. Remove the sample from the sampler by pushing or scraping the soil with an appropriate precleaned utensil into an appropriately precleaned tray or aluminum foil.
- 3.5. Remove any portion of the sample that has been disturbed and discard.
- 3.6. Follow the sample collection procedures outlined in FS 3210.

NOTE: If a confining layer has been breached during sampling, grout the hole to land surface with Type-1 Portland cement. This requirement may be different throughout Florida; contact the local Water Management District office for local requirements.

4. SPLIT SPOON SAMPLER: Suitable for reaching soils from approximately 12 inches to depths greater than 10 feet.

- 4.1. A split spoon sampler, useful for sampling unconsolidated soil, consists of two half cylinders (spoons) that fit together to form a tube approximately two feet in length and two inches in diameter.
 - 4.1.1. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at each end of the split spoon.
 - 4.1.2. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.
 - 4.1.3. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.
 - 4.1.4. Insert a catcher device in the head ring to prevent loss of unconsolidated sample during recovery.
- 4.2. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.
- 4.3. If the recovery is enough to accommodate discarding a portion of the sample, discard the top and bottom two to three inches of the sample.
- 4.4. For volatile organic compounds collect the sample immediately from the **center portion of the split spoon** using the procedures described in FS 3000, section 5.
- 4.5. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.
- 4.6. Select an appropriate precleaned sampling device and collect the sample.
- 4.7. Transfer the sample to the appropriate sample container.
- 4.8. Clean the outside of the sample container to remove excess soil.
- 4.9. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

5. DIRECT PUSH RIGS: May be used for depths greater than 10 feet below ground surface.

- 5.1. Liners: The clear liners are used with direct push rigs. This method is appropriate only for unconsolidated materials. The sampling depth that can be achieved varies depending on the rig and the lithologies that are encountered. Typically, the rig operator will:

- Place the liner inside the metal probe rod
- Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod
- Advance the rod a full rod length
- Retrieve the rod
- Remove the point holder
- Remove the liner, and
- Slice the liner to expose the soil.

5.2. After the liner has been sliced, follow the procedures outlined in FS 3210, collecting volatile organic samples (if needed) immediately after the liner is sliced.

5.3. If samples for organic vapor analysis screening are required, collect them by slicing the sample(s) using a clean, decontaminated utensil and place them in 8-ounce (preferred) or 16-ounce jars, immediately cover the opening with aluminum foil and screw on the lid ring. If the contamination is derived from petroleum products, it is acceptable to use a clean gloved hand to transfer the sample(s) to the sample container(s).

5.4. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

5.5. Select an appropriate precleaned sampling device and collect the sample.

5.6. Transfer the sample to the appropriate sample container.

5.7. Clean the outside of the sample container to remove excess soil.

5.8. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

6. SHELBY TUBE SAMPLER

6.1. The Shelby tube sampler is used to sample unconsolidated soil and consists of a tube approximately 30 inches long and two inches (or larger) in diameter.

6.2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter, which allows attachment to the drilling rig assembly.

6.3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.

6.4. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or manually with a sledge hammer.

6.5. Remove the tube from the sampler head.

6.6. Extrude the sample from the Shelby tube.

6.7. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

6.8. Collect samples for volatile organics immediately from the center portion of the Shelby tube using the procedures described in FS 3000, section 5.

6.9. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.

- 6.10. Transfer the sample to the appropriate sample container.
- 6.11. Clean the outside of the sample container to remove excess soil.
- 6.12. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

7. CORE BARREL

- 7.1. A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.
 - 7.1.1. The core barrel is a cylinder approximately three feet long and two inches in diameter.
 - 7.1.2. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soil as the drilling rods are rotated.
- 7.2. Retrieve the sample core by unscrewing the head ring and sliding the sample into a precleaned container.
- 7.3. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.
- 7.4. Remove the sample from the sampler (corer) with a precleaned tool.
- 7.5. Transfer the sample to the appropriate sample container.
- 7.6. Clean the outside of the sample container to remove excess soil.
- 7.7. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FT 1000. GENERAL FIELD TESTING AND MEASUREMENT

Use the following SOPs in conjunction with FT 1000:

- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FS 1000 General Sampling Procedures
- FT 1100 through FT 3000 Specific Field Testing Procedures

1. INTRODUCTION

1.1. Scope and Applicability: SOPs FT 1100 to FT 3000 outline procedures to conduct field testing measurements and observations. They include the parameters that are measured *in-situ* or in a field-collected sample. Additionally some samples with allowable extended holding times may be collected for laboratory measurement, as described in the specific FT-series SOPs. Included in SOPs FT 1100 to FT 3000 are:

- FT 1100 Field Measurement of Hydrogen Ion Activity (pH)
- FT 1200 Field Measurement of Specific Conductance (Conductivity)
- FT 1300 Field Measurement of Salinity
- FT 1400 Field Measurement of Temperature
- FT 1500 Field Measurement of Dissolved Oxygen (DO)
- FT 1600 Field Measurement of Turbidity
- ~~• FT 1700 Field Measurement of Light Penetration (Secchi Depth and Transparency)~~
- ~~• FT 1800 Field Measurement of Water Flow and Velocity~~
- ~~• FT 1900 Continuous Monitoring with Installed Meters~~
- ~~• FT 2000 Field Measurement of Residual Chlorine~~
- ~~• FT 3000 Aquatic Habitat Characterization~~

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1.2. Exclusions: **If proposed for experimental purposes, field-screening procedures employing techniques not addressed in these SOPs** must be submitted to the DEP site or project manager. Such procedures must be addressed for each program or project dealing specifically with the planning and design of sampling events. Data quality objectives for quantitative assessment preclude the use of field-screening procedures for regulatory purposes.

1.3. Expectations and Requirements:

1.3.1. In some cases, specific instruments are identified in the SOP, with detailed instruction provided on their use. If you are using a different instrument from that identified in the SOP, follow the manufacturer's instructions for assembly, operation, and maintenance.

1.3.2. When required, the FT-series SOPs outline the instrument specifications. A field instrument must meet the stated requirements.

1.3.3. The FT-Series SOPs specify the calibration requirements for each method. Although instruments may vary in configuration or operation, the specified calibration requirements must be met.

1.3.3.1. Where applicable to the FT-series SOP, use the minimum number of calibration standards specified.

1.3.3.2. Do not establish the lower limit of the quantitative calibration bracket with "zero" solutions, quality control blanks or reagent dilution water.

1.3.4. Ensure that all equipment is in proper working condition, calibrated, and that batteries are properly charged before using the equipment for field testing measurements.

1.3.5. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better. Some procedures may specify a higher grade or assay of reagent or standard.

1.4. Recommendations for Use of Grab Samples or *in situ* Field Testing Measurements:

1.4.1. Use *in situ* readings where practical for field measurements in surface water and wastewater.

1.4.2. Use *in situ* readings or flow-through containers for field measurements for groundwater stabilization during purging and for other applications where groundwater monitoring measurements are required.

1.4.3. If grab samples are collected for measurement where allowed in the individual FT-series SOP, measure samples within fifteen (15) minutes of collection when immediate analysis is specified per Table FS 1000-4 and FS 1000-5. Otherwise, analyze grab samples within the applicable holding times specified in Table FS 1000-4 and FS 1000-5.

2. MINIMUM CALIBRATION REQUIREMENTS:

2.1. Calibration Definitions: This section outlines the essential calibration concepts that must be applied to each field test. Specific requirements for calibration are addressed in the individual SOPs.

2.1.1. Initial Calibration (IC): The instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., dissolved oxygen saturation) or a known value of a calibration standard.

2.1.2. Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following initial calibration by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.3. Continuing Calibration Verification (CCV): The instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.4. Chronological Calibration Bracket: The interval of time between verifications within which environmental sample measurements must occur. The instrument or meter

is calibrated or verified before and verified after the time of environmental sample measurement(s).

2.1.5. Quantitative Calibration Bracket: The instrument or meter is calibrated or verified at two known values that encompass the range of observed environmental sample measurement(s).

2.1.6. Acceptance Criteria: The numerical limits within which calibration verifications are acceptable.

2.2. Calibration Activities: Specific calibration procedures are given in the individual SOPs.

2.2.1. Chronological Calibration Bracket:

2.2.1.1. Ensure that the field test result is preceded by an acceptable ICV or CCV and followed by an acceptable CCV.

2.2.1.2. Specific requirements for chronological bracketing are addressed in the individual FT-series SOPs.

2.2.2. Quantitative Calibration Bracket:

2.2.2.1. Choose two standards that bracket the range of sample measurements. These standards may be used for initial calibrations or for verifications.

2.2.2.2. Specific requirements for quantitative bracketing are addressed in the individual FT-series SOPs.

2.2.3. Initial Calibration: Calibrate if no initial calibration has been performed or if a calibration verification does not meet acceptance criteria. Do not reuse standards for initial calibrations.

Table FT 1000-1: Field Testing Acceptance Criteria	
Parameter	Acceptance Criteria
pH (FT 1100)	± 0.2 Standard pH Units of buffer or more stringent program criteria
Specific Conductance (FT 1200)	± 5% of standard value
Temperature (FT 1400)	± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values
Dissolved Oxygen (FT 1500)	± 0.3 mg/L of theoretical value (see Table FT 1500-1)
Turbidity (FT 1600)	0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value
Total Residual Chlorine (FT 2000)	0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value

2.2.4. Initial Calibration Verification:

2.2.4.1. Perform an ICV immediately after calibration. All ICVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.4.2. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

2.2.5. Continuing Calibration Verification: Perform a CCV at no more than 24-hour intervals from previous verification, except where noted for individual FT-series SOPs.

2.2.5.1. If historically generated data demonstrate that a specific instrument remains stable for longer periods of time, the time interval between calibration verifications may be increased.

2.2.5.2. Base the selected time interval on the shortest interval that the instrument maintains stability. If CCVs consistently fail, shorten the time period between verifications or replace/repair the instrument.

2.2.5.3. All CCVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.5.4. If a CCV fails to meet acceptance criteria perform one or more of the following procedures as necessary:

- Reattempt the CCV again within the chronological bracket time interval without changing the instrument calibration. Do not perform maintenance, repair, or cleaning of the instrument or probe. Probes may be rinsed with analyte-free water or fresh verification standard. The CCV may be reattempted with a fresh aliquot of verification standard.
- Perform the initial calibration, perform an ICV, re-analyze the sample(s), and perform a CCV.
- Report all results between the last acceptable calibration verification and the failed calibration verification as estimated (report the value with a "J"). Include a narrative description of the problem in the field notes.

2.2.5.5. For installed instruments that are deployed for extended periods of time or used for continuous monitoring, see FT 1900.

2.2.5.6. Shorten the time period between verification checks or replace/repair the instrument.

2.2.6. Determining the Values of Secondary Standards: Use only those standards recommended by the manufacturer for a specific instrument. Only use secondary standards for continuing calibration verifications. See the individual FT-series SOPs for specific procedures for use of secondary standards. At documented intervals, determine or verify the values of secondary standards immediately after performing an initial calibration or after verifying the calibration with primary standards. Read each secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10% of the stated standard value. If the +/- 10% criterion is not

met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

2.2.7. More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.

3. PREVENTIVE MAINTENANCE: Record all maintenance and repair notes in the maintenance logbook for each meter (see FS 1007). If rental equipment is used, a log is not required. However, the origin (i.e., rental company), rental date, equipment type, model number, and identification number (if applicable) must be entered into the field notes or a rental equipment notebook.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Manufacturer's tolerance range for secondary standards
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document the date and time of any corrective actions.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1000
Tables, Figures and Forms

Table FT 1000-1 Field Testing Acceptance Criteria

Table FT 1000-1: Field Testing Acceptance Criteria	
Parameter	Acceptance Criteria
pH (FT 1100)	± 0.2 Standard pH Units of buffer or more stringent program criteria
Specific Conductance (FT 1200)	$\pm 5\%$ of standard value
Temperature (FT 1400)	$\pm 0.2^{\circ}\text{C}$ of NIST-traceable value (with correction factors) Verification over range of applicable values
Dissolved Oxygen (FT 1500)	± 0.3 mg/L of theoretical value (see Table FT 1500-1)
Turbidity (FT 1600)	0.1-10 NTU: $\pm 10\%$ of standard value 11-40 NTU: $\pm 8\%$ of standard value 41-100 NTU: $\pm 6.5\%$ of standard value > 100 NTU: $\pm 5\%$ of standard value
Total Residual Chlorine (FT 2000)	0.995 calibration curve correlation coefficient $\pm 10\%$ of primary standard value $\pm 10\%$ of secondary standard value Color comparator acceptance criterion: $\pm 10\%$ of primary standard value

FT 1100. Field Measurement of Hydrogen Ion Activity (pH)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. Equipment and Supplies

1.1. Field Instrument: Use any pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device.

1.1.1. For routine fieldwork use a pH meter accurate and reproducible to at least 0.2-unit in the range of 0.0 to 14.0 units, and equipped with temperature-compensation adjustment. Record the pH value in pH units to one decimal place.

1.1.2. Advanced silicon chip pH sensors (with digital meters) may be used if demonstrated to yield equivalent performance to glass electrode sensors for the intended application.

1.2. Standards: Purchased or laboratory-prepared standard buffer solutions of pH values that bracket the expected sample pH range. Use buffers with nominal values of 4.0, 7.0 and 10.0 units for most situations. If the sample pH is outside the range of 4.0 to 10.0, then use two buffers that bracket the expected range with the pH 7 buffer being one of the two buffers. Alternatively, prepare appropriate standards per table I in method SM4500-H⁺-B.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. Calibration and Use

2.1. General Concerns

2.1.1. The acceptance criterion for the initial calibration or the calibration verification is a reading of the standard within +/- 0.2-unit of the expected value.

2.1.2. On a weekly basis, check the calibration to ensure the % theoretical slope is greater than 90% (if applicable to your instrument type).

2.1.2.1. Note the % slope in the calibration records.

2.1.2.2. A % slope of less than 90% indicates a bad electrode that must be changed or repaired.

2.1.2.3. If % slope cannot be determined on your meter, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

2.2. Interferences

2.2.1. Sodium at pH \geq 10.0 units can be reduced or eliminated by using a low sodium error electrode.

- 2.2.2. Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use acetone very sparingly so that the electronic surface is not damaged.
- 2.2.3. Temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples.
- 2.2.4. Poorly buffered solutions with low specific conductance ($< 200 \mu\text{mhos/cm}$) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.
- 2.2.5. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.
- 2.2.6. Thoroughly rinse the pH sensor with deionized water or fresh buffer standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or standards of widely different pH value are successively measured.
- 2.2.7. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrode per the manufacturer's instructions or replace.
- 2.3. Calibration: Follow the manufacturer's calibration instructions specific to your meter. Most instruments allow for a two-point calibration and a few models can perform a three-point calibration. Use the appropriate number of standard buffer solutions for calibration. Do not reuse buffers for initial calibrations.
 - 2.3.1. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution.
 - 2.3.2. Follow the calibration activities specified in FT 1000, section 2.2.
 - 2.3.2.1. Perform an initial calibration using at least two buffers. Always use a pH 7 buffer first.
 - 2.3.2.2. If the pH sample range is expected to be wider than the range established by a two-point calibration (e.g., some samples at pH 4 and others at pH 8), then add a third calibration point. If the instrument cannot be calibrated with three buffers, the third buffer may be used as the initial calibration verification to extend the range.
 - 2.3.2.3. After initial calibration, immediately perform an initial calibration verification (ICV). Read a buffer as a sample. To be acceptable, a calibration verification must be within ± 0.2 pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range. Certain regulatory programs may have more stringent acceptance criteria.
 - 2.3.2.4. After sample measurement(s), perform a continuing calibration verification (CCV). Read a buffer as a sample. To be acceptable, a

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calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

- 2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.
- 2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.
- 2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).
 - 2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.
 - 2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.
 - 2.6.3. Wait for stabilization, and read the pH value.
 - 2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.
3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.
4. DOCUMENTATION
 - 4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.
 - 4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.
 - 4.1.1.1. Document acceptable verification of any standard used after its expiration date.
 - 4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.
 - 4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.
 - 4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.
 - 4.1.3. Record the grade of standard or reagent used.
 - 4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.
 - 4.1.4.1. Record the date of preparation for all in-house formulations.
 - 4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).
 - 4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

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- 4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.
 - 4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.
 - 4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
 - 4.2.3. Record the time and date of all initial calibrations and all calibration verifications.
 - 4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
 - 4.2.5. Record the name of the analyst(s) performing the calibration.
 - 4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
 - Type of standard or standard name (e.g., pH buffer)
 - Value of standard, including correct units (e.g., pH = 7.0 SU)
 - Link to information recorded according to section 4.1 above
 - 4.2.7. Retain manufacturers' instrument specifications.
 - 4.2.8. Document whether successful initial calibration occurred.
 - 4.2.9. Document whether each calibration verification passed or failed.
 - 4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
 - 4.2.10.1. Document date and time of any corrective action.
 - 4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
 - 4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 4.3. Record all field-testing measurement data, to include the following:
- Project name
 - Date and time of measurement or test (including time zone, if applicable)
 - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
 - Latitude and longitude of sampling source location (if required)
 - Analyte or parameter measured
 - Measurement or test sample value
 - Reporting units
 - Initials or name of analyst performing the measurement
 - Unique identification of the specific instrument unit(s) used for the test(s)

FT 1200. Field Measurement of Specific Conductance (Conductivity)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.5. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100 $\mu\text{mhos/cm}$ or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100 $\mu\text{mhos/cm}$ standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100 $\mu\text{mhos/cm}$.

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100 $\mu\text{mhos/cm}$, only one standard at 100 $\mu\text{mhos/cm}$ standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100 $\mu\text{mhos/cm}$ standard is 95 to 105 $\mu\text{mhos/cm}$. If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using

the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}$$

Where: K = conductivity in $\mu\text{mhos/cm}$ at 25°C

K_m = measured conductivity in $\mu\text{mhos/cm}$ at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}$$

Refer to SM2510B, 20th edition, if other calculations (i.e., determining cell constant, etc.) are required. See FT 1210 below.

3.5 *In situ* Measurements at Depth or With Flow-through Cells: After calibrating the instrument as outlined in 3.2 above, follow the manufacturer's instructions to measure the conductivity of the sample.

3.5.1. For *in situ* measurements immerse the probe at the desired depth and wait for stabilization of the reading and record its value. Follow a similar procedure when using a flow-through cell.

3.5.1.1 Preferably measure groundwater sample conductivity *in situ* with a downhole probe or in a flow-through system.

4. PREVENTATIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

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- 5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
- 5.2.3. Record the time and date of all initial calibrations and all calibration verifications.
- 5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
- 5.2.5. Record the name of the analyst(s) performing the calibration.
- 5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
 - Type of standard or standard name (e.g., conductivity standard)
 - Value of standard, including correct units (e.g., conductivity = 100 µmhos/cm)
 - Link to information recorded according to section 5.1 above
- 5.2.7. Retain manufacturers' instrument specifications.
- 5.2.8. Document whether successful initial calibration occurred.
- 5.2.9. Document whether each calibration verification passed or failed.
- 5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
 - 5.2.10.1. Document date and time of any corrective action.
 - 5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 5.3. Record all field-testing measurement data, to include the following:
 - Project name
 - Date and time of measurement or test (including time zone, if applicable)
 - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
 - Latitude and longitude of sampling source location (if required)
 - Analyte or parameter measured
 - Measurement or test sample value
 - Reporting units
 - Initials or name of analyst performing the measurement
 - Unique identification of the specific instrument unit(s) used for the test(s)

FT 1400. Field Measurement of Temperature

The use of this SOP is not required when using field temperature measurement devices to monitor groundwater stabilization during the purging of groundwater monitoring wells. Field temperature measurement devices used for temperature compensation (correction) for other measurements such as dissolved oxygen, specific conductance or pH are also exempted from the requirements of this SOP. FT 1400 must be used for all other field temperature measurements required by DEP.

Use this SOP in conjunction with the following DEP SOPs:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments: Use any of the following instrument types for performing field measurements:

- Digital thermistor (thermocouple type) and meter typical of field instruments
- Glass bulb, mercury-filled thermometer (not recommended for field ruggedness)
- Glass bulb, alcohol-filled thermometer with protective case
- Bi-metal strip/dial-type thermometer
- Advanced silicon chip temperature sensor and digital meter

1.1.1. Field instruments must be capable of measuring temperature in 0.1°C increments.

1.2. Standard Thermometer: NIST-traceable Celsius certified thermometer with scale marks for every 0.1°C increment, a range of 0°C to 100°C (or a range bracketing expected sample temperatures) and correction chart supplied with certification. The standard thermometer must have a valid certification for the period of measurement.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook or forms \
- Indelible pens

2. CALIBRATION AND USE

2.1. General Concerns

2.1.1. Select a temperature measuring device meeting the requirements of section 1.1 above.

2.1.2. Dial-type and thermocouple-type devices with meters are preferred over the glass thermometers for fieldwork because of their durability and ease of reading.

2.1.2.1. Transport glass thermometers in protective cases.

2.1.2.2. Inspect glass thermometers for liquid separation. Do not use a thermometer if the liquid has separated.

2.1.2.3. Most instruments with digital display will provide more decimal figures than are significant. Record the temperature reading with only one rounded decimal figure (e.g., 25.9 instead of 25.86°C).

2.2. Calibration

2.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

2.2.2. Verify all thermistor (meter) devices and field thermometers against the NIST-traceable standard thermometer at several temperatures in the expected sample measurement range, using any correction factor indicated by the certificate supplied with the NIST-traceable thermometer.

2.2.2.1. See the US Geological Survey, National Field Manual for the Collection of Water-Quality Data, Book 9, Chapter A6, Field Measurements, Section 6.1, Temperature, Techniques of Water-Resources Investigations, 4/98 for additional guidance about making temperature comparisons with the standard thermometer.

2.2.2.2. Make note of the calibration in the calibration records. See section 4 below.

2.2.2.3. The field measurement device may be used with a linear correction factor provided that the observed temperature difference with the standard thermometer is documented at incremental temperatures over the range of expected sample temperatures.

2.2.2.4. Use the resulting correction factor when making temperature measurements of samples with the field measurement device.

2.2.2.5. Prominently display the correction factor on the field measurement device, with the date last verified. A calibration correction curve or plot may also be used.

2.2.2.6. To be acceptable, a calibration verification must be within +/- 0.5°C of the corrected reading of the NIST-traceable thermometer.

2.2.2.7. Properly dispose of glass-bulb thermometers that do not meet the above calibration acceptance criteria.

2.2.3. Continuing Calibration Verifications:

2.2.3.1. Determine the maximum time between continuing calibration verifications for the specific field temperature measurement device based on instrument stability.

2.2.3.2. Verify the field measurement device against the standard NIST-traceable thermometer as in section 2.2.2 above.

2.2.4. Refer to additional calibration requirements in FT 1000, section 2.2.

2.2.5. More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.

2.3. Measuring Sample Temperature

2.3.1. Insert or place the thermometer or sensor *in situ* at a measuring location representative of the sampling source.

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2.3.2. Allow the thermometer or temperature sensor to equilibrate to ambient *in situ* temperature.

2.3.2.1. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.3.3. Record the temperature to the nearest 0.1°C after the reading stabilizes and remains constant.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standards Documentation: Document information about the NIST-traceable standard thermometer in the calibration record, including:

- Unique identification for the thermometer
- Vendor certificate of calibration, including any correction factor
- Vendor's expiration date for the certificate of calibration

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the following information about initial calibration and calibration verifications and link to information recorded according to section 4.1 above:

- Details of the method used to compare the field measurement device to the NIST-traceable standard thermometer.
- Results of each calibration verification, including the expected reading (per the NIST-traceable standard thermometer)
- The actual reading of the field measurement device, using any established correction factors and correct units.

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance (such as a new correction factor) according to records requirements of FD 3000.

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- 4.2.10.1. Document date and time of any corrective action.
- 4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 4.3. Record all field-testing measurement data, to include the following:
 - Project name
 - Date and time of measurement or test (including time zone, if applicable)
 - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
 - Latitude and longitude of sampling source location (if required)
 - Analyte or parameter measured
 - Measurement or test sample value
 - Reporting units
 - Initials or name of analyst performing the measurement
 - Unique identification of the specific instrument unit(s) used for the test(s)

FT 1500. Field Measurement of Dissolved Oxygen (DO)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments

1.1.1. Membrane-type polarographic or galvanic electrode DO sensor with dedicated meter or configured with multi-parameter sonde

1.1.2. Luminescence-based DO sensor with dedicated meter or configured with multi-parameter sonde (see American Society for Testing and Materials, *Standard Test Methods for Dissolved Oxygen in Water*, Test Method C-Luminescence-based Sensor, D 888-05).

1.1.3. Select instrument assemblies that provide minimum precision of +/- 0.2 mg DO/L and a minimum accuracy of +/- 0.2 mg DO/L.

1.1.4. Compensate for temperature dependence of DO measurements by using instruments employing automatic temperature compensation or by manually correcting measurements in accordance with SM 4500-O G (see *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, American Water Works Association, Water Pollution Control Federation).

1.1.4.1. Calibrate on-board temperature sensors as described in FT 1400.

1.2. Standards

1.2.1. NIST-traceable Celsius thermometer with a scale marked for every 0.1°C and a range of 0 to 100°C.

1.2.2. Access to an organization with capability to perform the Winkler titration procedure is recommended but not mandatory.

1.2.3. A “zero-DO standard”, prepared on-site with an aliquot of the sample water, is optional. Prepare by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. CALIBRATION AND USE: the electrode method is predominantly used in-situ for dissolved oxygen determinations.

2.1. General Concerns

2.1.1. Turbulence is necessary to keep a constant flow of water across the membrane-sample interface. Make sure the appropriate mechanism is working before using the probe.

2.1.2. Follow instrument manufacturer's instructions for probe storage. For example, store the probe with a cover that creates a saturated atmosphere. A cap, with a wet sponge in it, will suffice for single-parameter probes. If the sensor is in a multi-probe device, keep the protective cap chamber moist during storage.

2.1.3. Before mobilizing, check to make sure there are no bubbles beneath the probe membrane, or any wrinkles or tears in the probe membrane. If so, replace the membrane and KCL solution. Check the leads, contacts, etc. for corrosion and/or shorts if meter pointer remains off-scale, does not calibrate, or drifts.

2.1.4. Dissolved inorganic salts interfere with the performance of DO probes. For example, DO readings in salt water are affected by the salinity and must be corrected. The DO meter may adjust automatically based on readings taken from the specific conductivity/salinity probe. If corrections are not automatic the appropriate calculations must be used to correct for salinity. If automatic adjustments are used the specific conductivity/salinity probe calibration must be verified or calibrated in accordance with FT1200.

2.1.5. Reactive gases, which pass through the membrane, may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Sulfide (from H₂S) will undergo oxidation if high enough potential (voltage) is applied, creating current flow, yielding faulty readings. If such interferences are suspected, change the membrane electrode more frequently and calibrate at more frequent intervals.

2.1.6. Ensure that the temperature of the sensor and sample are stable. Unstable temperatures will produce erroneous calibrations, verifications or sample measurements.

2.1.7. Erroneous calibrations or verifications may result if the saturated air chamber is not vented to atmospheric pressure, properly humidified and protected from temperature fluctuations produced by common field conditions such as evaporation or fluctuation in sunlight intensity.

2.2. Follow the quality control requirements for calibration (see activities in FT 1000, section 2.2).

2.3. Initial Calibration and Initial Calibration Verification

2.3.1. Air Calibration and Initial Calibration Verification (ICV): Calibrate the meter at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument.

2.3.1.1. Allow an appropriate warm up period before initial field calibration.

2.3.1.2. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100% humidity).

2.3.1.3. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.3.1.4. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table, what the DO saturation value should be at the observed temperature (see Table FT

1500-1, below). A stable and accurate temperature is required for a valid calibration. The acceptance criterion for DO calibration verification is +/- 0.3 mg DO/L at the observed temperature of the verification.

2.4. Continuous Calibration Verification

2.4.1. Air-Calibration Verification: DO sensor or instrument is calibrated against air that is saturated with water at a known temperature and ambient atmospheric pressure. Use Table FT 1500-1 below to verify calibration at specified temperature.

2.4.1.1. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops) and insert the sensor into the chamber (this ensures 100-percent humidity)

2.4.1.2. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.4.1.3. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes.

2.4.1.4. Use the oxygen solubility Table FT 1500-1 below to determine the DO saturation at a measured temperature and atmospheric pressure. Calculate values to the nearest tenth degree by interpolation or use an expanded version of this table found in FS 2200, which provides saturation data in 0.1 °C increments for a selected temperature range (see Table FS 2200-2).

2.4.1.5. Compare DO meter reading with value obtained from Table FT 1500-1 below to verify continuous calibration.

2.5. Additional Verifications: The following methods may be used as additional checks to verify calibration. These additional checks may be required as part of a specific permit.

2.5.1. Winkler method: This check is useful to assess the condition of the DO sensor (i.e., its degradation with time/use) and that the instrument can still maintain a valid calibration (see SM 4500-O C).

2.5.1.1. **Perform the Winkler method when required by permit or other regulation at the required calibration agency.**

2.5.1.2. For an accuracy calibration using the Winkler method, follow SM 4500-O C.

2.5.1.3. Fill a clean bucket with uncontaminated or de-ionized water and place the probe into the bucket (with stirrer or equivalent mechanism turned off). Fill at least two biological oxygen demand (BOD) bottles without entraining atmospheric oxygen into the bottles. Carefully submerge the bottom of the bottle (one at a time) into the water and allow the water to fill the bottle. Place the bottle on the bottom of the bucket and carefully place stopper into it without adding atmospheric oxygen. Retrieve the bottles and determine their DO by the Winkler method (see SM4500-O-C for more details). Turn the stirrer or equivalent mechanism on and read the DO of the water in the bucket.

2.5.1.4. Adjust the DO meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of water in the bucket, and then calibrate the DO meter to read the average DO concentration of the two samples determined by the Winkler test.

~~2.5.2. Zero-DO Verification: The air calibration and the interfering effects of the sample can be further checked in the field by means of a "zero-DO standard"(SM 4500-O G).~~

~~2.5.2.1. Prepare this standard on-site with an aliquot of the sample by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero. Prepare this zero-DO standard in a beaker or a large-mouth sample container of appropriate size to insert the DO probe.~~

~~2.5.2.2. After adding the chemicals, gently swirl the water and let it sit for about 30 seconds before inserting the probe.~~

~~2.5.2.3. Read the DO of the sample. If the reading is outside the acceptance interval, the instrument must be recalibrated and/or zero-adjusted if the meter allows for this adjustment.~~

2.5.3. Air-Saturated Water: The DO sensor TD
7/20/11 ent system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

2.5.3.1. The temperature and conductivity of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

2.5.3.2. Place DO sensor and calibration water in a large beaker or open-mouth container.

2.5.3.3. Aerate the water for an adequate amount of time.

2.5.3.4. Determine if the water is 100 percent saturated with oxygen, and take a temperature reading. Temperature must be calibrated or verified for accuracy before DO calibration verification.

2.5.3.5. Use Table FT 1500-1 above to determine the DO saturation value at the measured water temperature. Compare DO meter reading with value obtained from Table FT 1500-1 to ensure continuous calibration.

2.6. Measuring DO in Samples:

2.6.1. Insert or place the DO probe *in situ* at a measuring location representative of the sampling source:

2.6.1.1. Take the DO of an effluent just before it enters the receiving water. If the effluent aerated prior to entering the surface water, take the DO reading in the receiving water right where it enters.

2.6.1.2. For well mixed surface waters, e.g., fast flowing streams, take the DO reading at approximately 1-2 feet below the surface or at mid-depth.

2.6.1.3. For still or sluggish surface waters, take a reading at one foot below the surface, one foot above the bottom, and at mid-depth.

2.6.1.4. If it is shallow surface waters, (less than two feet) take the reading at mid-depth.

2.6.1.5. Do not take a reading in frothy or aerated water unless required by the sampling plan.

2.6.1.6. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.6.2. Rinse probe with de-ionized water and keep the probe in the saturated atmosphere (see 2.1.2 above) between sites and events.

2.6.3. If the readings show distinct, unexplainable changes in DO levels, or when the probe has been in waters with high sulfides, recalibrate or perform maintenance per manufacturer's instructions. While taking a reading, if it is very low (e.g., below 1.0 mg/L), allow the meter to stabilize, record it and then, remove and rinse the probe, as the environment is very likely anoxic and may contain hydrogen sulfide, which can damage the probe.

2.6.4. Salinity and Temperature corrections may be necessary. Follow manufacturer instructions for automatic corrections or perform manual calculations (SM 4500-O G).

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for verifications.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

- 4.2.5. Record the temperature associated with all calibration verifications.
- 4.2.6. Record the name of the analyst(s) performing the calibration.
- 4.2.7. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
 - Type of standard or standard name (e.g., saturation)
 - Value of standard, including correct units (e.g., mg/L at °C)
 - Link to information recorded according to section 4.1 above
- 4.2.8. Retain manufacturers' instrument specifications.
- 4.2.9. Document whether successful initial calibration occurred.
- 4.2.10. Document whether each calibration verification passed or failed.
- 4.2.11. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
 - 4.2.11.1. Document the date and time of any corrective action.
 - 4.2.11.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 4.2.12. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 4.3. Record all field-testing measurement data, to include the following:
 - Project name
 - Date and time of measurement or test (including time zone, if applicable)
 - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
 - Latitude and longitude of sampling source location (if required)
 - Analyte or parameter measured
 - Measurement or test sample value
 - Reporting units
 - Initials or name of analyst performing the measurement
 - Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1500
Tables, Figures and Forms

Table FT 1500-1 Solubility of Oxygen in Water

Table FT 1500-1: Solubility of Oxygen in Water			
at Atmospheric Pressure^{1,2}			
Temperature	Oxygen Solubility	Temperature	Oxygen Solubility
°C	mg/L	°C	mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

1. The table provides three decimal places to aid interpolation
2. Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water-saturated

FT 1600. Field Measurement of Turbidity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Turbidity measures the scattering effect that suspended solids have on the propagation of light through a body of water (surface or ground waters). The higher the effect (i.e., intensity of scattered light), the higher the turbidity value. Suspended and colloidal matter such as clay, silt, finely divided organic and inorganic matter, and plankton and other microscopic organisms cause turbidity in water.

This SOP describes the use of true nephelometric measurement using instruments meeting the specifications outlined in 2.1.

Exceptions to the requirements specified in 2.1 below include:

- 1.1. In situ probes with turbidity sensors used for screening purposes (e.g., groundwater purge stabilization measurements).
- 1.2. Non standard light sources, detectors or other turbidity measuring devices may be proposed for use in studies that entail comparison measurements (dredge and fill) or unattended deployment for monitoring purposes.
- 1.3. **Do not report results from “non standard” sensors or configurations for regulatory purposes such as permit compliance unless the Department has approved the use for the specific project.**
- 1.4. All “non standard” instrument must be calibrated/check according to the principles outlined in this SOP.

2. EQUIPMENT AND SUPPLIES

- 2.1. Field Instrument: Use a turbidimeter (nephelometer) or a spectrophotometer consisting of a light source and one or more photoelectric detectors with a readout device to indicate the intensity of light. The instrument must meet these specifications:
 - 2.1.1. The light source must have a tungsten-filament lamp operated at a color temperature between 2000 and 3000 K.
 - 2.1.2. The distance traversed by the incident light and scattered light within the sample tube must not exceed 10 cm.
 - 2.1.3. The light detector, positioned at 90° to the incident light, must have an acceptance angle that does not exceed $\pm 30^\circ$ from 90°.
 - 2.1.4. The detector and any filter system must have a spectral peak response between 400 and 600 nanometers.
 - 2.1.5. The instrument sensitivity must permit detection of a turbidity difference of 0.02 NTU at the 0 – 1.0 NTU scale.

2.1.6. Note: using the appropriate equipment and following the procedures in this SOP, the field accuracy of this measurement is close to $\%R = 100 \pm 10\%$ for turbidities in the range of 1 to 100 NTU.

2.2. Sample Cells (cuvettes): Use sample cells or tubes of clear, colorless glass or plastic.

2.2.1. Keep cells clean, both inside and out, and discard if scratched or etched.

2.2.1.1. Never handle them where the light beam strikes the sample.

2.2.1.2. Clean sample cells by thorough washing with laboratory soap (inside and out) followed by multiple rinses with distilled or de-ionized water, and let air-dry.

2.2.2. Use a very thin layer of silicone oil on the outside surfaces to mask minor imperfections or scratches in the cells.

2.2.2.1. Use silicone oil with the same refractive index of the glass; making sure the cell appear to be nearly dry with little or no visible signs of oil.

2.2.3. Because small differences between cells significantly impact measurement, use either matched pairs or the same cell for standardization and sample measurement.

2.3. Standards:

2.3.1. Primary standards: Use these standards for initial calibration.

2.3.1.1. Formazin standards can be either obtained commercially or prepared according to method SM 2130B, section 3.b. See *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association, American Water Works Association, Water Pollution Control Federation).

2.3.1.2. Some instruments may require the use of styrene divinylbenzene (SDVB) standards for calibration.

2.3.2. Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards must only be used for continuing calibration verifications according to the procedures in section 3.4 below. Determine or verify the values of secondary standards according to the procedure in section 3.3 below.

2.3.3. Turbidity-free water: Use filtered, laboratory reagent water demonstrated to be free of measurable turbidity (<0.01 NTU) or purchase commercially prepared turbidity-free water.

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Light absorption by dissolved and suspended matter may cause a negative bias on the turbidity measurement. When present in significant concentrations, particles of light-absorbing materials such as activated carbon will cause a negative interference. Likewise, the presence of dissolved, color-causing substances that absorb light may also cause a negative interference. Some commercial instruments may have the capability of either correcting for slight color interference or optically blanking out the color effect.

3.1.2. Handle samples with natural effervescence as described in 3.5.5.1 below.

3.2. Calibration and Initial Calibration Verification

3.2.1. Follow the calibration activities in FT 1000, section 2.2.

3.2.2. Perform an initial calibration using at least two primary standards.

3.2.2.1. If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard per 3.2.3 below.

3.2.2.2. For measurement of samples of very low turbidity, select the lowest standard commercially available for bracketing the lower end of the anticipated sample turbidity range or dilute higher turbidity standards with turbidity-free water.

3.2.2.3. Do not use turbidity-free water as a calibration verification standard.

3.2.3. Perform an initial calibration verification by reading at least one primary standard as a sample. The acceptance criterion for the initial calibration verification depends on the range of turbidity of the standard value:

- Standard Value = 0.1-10 NTU: the response must be within 10% of the standard;
- Standard Value = 11-40 NTU: the response must be within 8% of the standard;
- Standard Value = 41-100 NTU: the response must be within 6.5% of the standard; and
- Standard Value > 100 NTU: the response must be within 5% of the standard.

3.3. Determining the Values of Secondary Standards

3.3.1. Use only those standards certified by the manufacturer for a specific instrument.

3.3.2. Use verified secondary standards only for continuing calibration verifications.

3.3.3. Determining the initial value(s) of secondary standard(s):

3.3.3.1. Calibrate or verify the instrument with primary standards. Select primary standards that bracket the range of the secondary standards.

3.3.3.2. Immediately after the an initial calibration with primary standards or verification with a primary standard, read each secondary standard as a sample use the reading from the instrument as the first assigned value.

3.3.4. Verifying Secondary Standards

3.3.4.1. At least once per quarter or at other documented intervals (see 3.3.5 below), determine or verify the values of secondary standards immediately after the instrument has been calibrated or verified with primary standards.

3.3.4.2. Read each secondary standard as a sample. This reading must be within the manufacturer's stated tolerance range and within the acceptance ranges of the assigned standard value as listed in 3.2.3., above. If the criteria in section 3.2.3., above are not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

3.3.5. More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.

3.4. Continuing Calibration Verification: Perform a continuing calibration verification using at least one primary or secondary standard. The calibration acceptance criteria are the same as those listed in section 3.2.3 above.

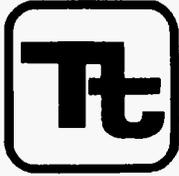
3.5. Measuring Turbidity in Samples

3.5.1. Gently agitate the sample and wait until air bubbles disappear.

- 3.5.2. Double-rinse the sample cell or cuvette with a small amount of the sample. Discard, and pour an aliquot into the sample cell or cuvette.
 - 3.5.3. Gently dry out its external surface with lint-free paper.
 - 3.5.4. Insert the cell in the instrument and read the turbidity directly from the meter display.
 - 3.5.5. Do not use vacuum degassing, ultrasonic bath or other devices to remove bubbles from the sample. If the sample contains visible bubbles or if it effervesces (as in groundwater, with changes in pressure and temperature), make a note of this in the field records and collect a sample for laboratory measurement.
 - 3.5.5.1. If effervescing samples are collected for laboratory analysis collect the sample without leaving headspace in the container and ship it as soon as possible to the laboratory (the holding time for this measurement is only 48 hrs). Ship this sample in wet ice at 4°C.
 - 3.5.6. Pour out the sample, double-rinse the cuvette with de-ionized water in preparation for the next sample.
4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.
 5. DOCUMENTATION
 - 5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.
 - 5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.
 - 5.1.1.1. Document acceptable verification of any standard used after its expiration date.
 - 5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.
 - 5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.
 - 5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.
 - 5.1.3. Record the grade of standard or reagent used.
 - 5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.
 - 5.1.4.1. Record the date of preparation for all in-house formulations.
 - 5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).
 - 5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.
 - 5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.
 - 5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

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- 5.2.2.1. Record manufacturer name, model number, and identifying number (such as a serial number) for each instrument unit.
- 5.2.3. Record the time and date of all initial calibrations and all calibration verifications.
- 5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
- 5.2.5. Record the name of the analyst(s) performing the calibration.
- 5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
 - Type of standard or standard name (e.g., formazin)
 - Value of standard, including correct units (e.g., 20 NTU)
 - Link to information recorded according to section 5.1 above
- 5.2.7. Retain manufacturers' instrument specifications.
- 5.2.8. Document whether successful initial calibration occurred.
- 5.2.9. Document whether each calibration verification passed or failed.
- 5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
 - 5.2.10.1. Document date and time of any corrective action.
 - 5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 5.3. Record all field-testing measurement data, to include the following:
 - Project name
 - Date and time of measurement or test (including time zone, if applicable)
 - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
 - Latitude and longitude of sampling source location (if required)
 - Analyte or parameter measured
 - Measurement or test sample value
 - Reporting units
 - Initials or name of analyst performing the measurement
 - Unique identification of the specific instrument unit(s) used for the test(s)



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>[Signature]</i>	

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT

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1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

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5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

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previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

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Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.
2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):
 - Well number.
 - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
 - Time and day of the measurement.
 - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

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5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

Pressure Transducer

TD
7/20/11

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

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5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

5.4 Equipment Decontamination

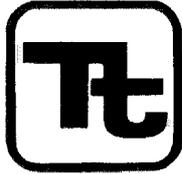
Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Effective Date 06/99	Revision 1
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject
BOREHOLE AND SAMPLE LOGGING

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

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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:

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 9 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 9 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 5 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'±				
	20.0			20.0					SET TEMP 6" CAS TO 15.5				
	21.0			21.0					SET 2"Ø PVC SCREEN 16-25	0	0	0	0
	22.0			22.0					SAND 14-25				
	23.0			23.0					PELLETS 12-14				

* When rock coring, enter rock brokenness.
 ** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.
 Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ± • 1-20Z
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z
NIX CORE IN BEDROCK RUN (1) = 25 min, RUN (2) = 15 min Drilling Area Background (ppm):
 Converted to Well: Yes No Well I.D. #: MW-1

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- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
 - Trace: 0 - 10 percent
 - Some: 11 - 30 percent
 - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
 - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
 - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
 - Particle shape - flat, elongated, or flat and elongated.
 - Maximum particle size or dimension.
 - Water level observations.
 - Reaction with HCl - none, weak, or strong.
- Additional comments:
 - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
 - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
 - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
 - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

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- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.
- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.
- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.
- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.
- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.
- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.
- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.
- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:
 - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
 - Indicate calcareous zones, description of any cavities or vugs.
 - Indicate any loss or gain of drill water.
 - Indicate drop of drill tools or change in color of drill water.
- Remarks at the bottom of Boring Log shall include:
 - Type and size of core obtained.
 - Depth casing was set.
 - Type of rig used.
- As a final check the boring log shall include the following:
 - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.
 - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

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5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

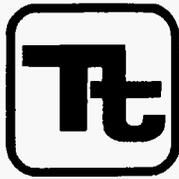
Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject
GROUNDWATER MONITORING WELL INSTALLATION

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1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

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5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

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The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

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Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

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5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium-to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

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A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized lid is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

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installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

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remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

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space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

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Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

- | | | | | |
|----|---------------------|--|---|------------------|
| 1 | Teflon® | | 5 | Lo-Carbon Steel |
| 2 | Stainless Steel 316 | | 6 | Galvanized Steel |
| 3. | Stainless Steel 304 | | 7 | Carbon Steel |
| 4 | PVC 1 | | | |

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton®*	Silicone	Neoprene	Teflon®*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

- | | | | | |
|----|------------------------|--|---|------------------------|
| 1 | Teflon® | | 5 | PE Conventional |
| 2 | Polypropylene (PP) | | 6 | Plexiglas/Lucite (PMM) |
| 3. | PVC Flexible/PE Linear | | 7 | Silicone/Neoprene |
| 4 | Viton® | | | |

* Trademark of DuPont

Source: Barcelona et al., 1983

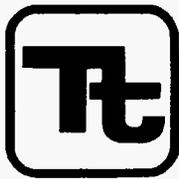
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ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Health & Safety		
Approved	D. Senovich <i>[Signature]</i>		

Subject
UTILITY LOCATING AND EXCAVATION CLEARANCE

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

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4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.
- 2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scars and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

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locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.
4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.
5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain
6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.
7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.
8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

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5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

<u>Nominal Voltage</u>	<u>Minimum Clearance</u>
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

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Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

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debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.
2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.
4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

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5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
 OSHA 29 CFR 1926(b)(2)
 OSHA 29 CFR 1926(b)(3)
 TtNUS Utility Locating and Clearance Policy
 TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
 TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
 TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

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**ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES**



American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

**ONE-CALL SYSTEMS INTERNATIONAL
CONDENSED DIRECTORY**

Alabama Alabama One-Call 1-800-292-8525	Iowa Iowa One-Call 1-800-292-8989	New Jersey New Jersey One Call 1-800-272-1000
Alaska Locate Call Center of Alaska, Inc. 1-800-478-3121	Kansas Kansas One-Call System, Inc. 1-800-344-7233	New Mexico New Mexico One Call System, Inc. 1-800-321-2537 Las Cruces- Dona Ana Blue Stakes 1-888-526-0400
Arizona Arizona Blue Stake 1-800-782-5348	Kentucky Kentucky Underground Protection Inc. 1-800-752-6007	New York Dig Safely New York 1-800-862-7962 New York City- Long Island One Call Center 1-800-272-4480
Arkansas Arkansas One Call System, Inc. 1-800-482-8998	Louisiana Louisiana One Call System, Inc. 1-800-272-3020	North Carolina The North Carolina One-Call Center, Inc. 1-800-632-4949
California Underground Service Alert North 1-800-227-2600 Underground Service Alert of Southern California 1-800-227-2600	Maine Dig Safe System, Inc. 1-888-344-7233	North Dakota North Dakota One-Call 1-800-795-0555
Colorado Utility Notification Center of Colorado 1-800-922-1987	Maryland Miss Utility 1-800-257-7777 Miss Utility of Delmarva 1-800-282-8555	Ohio Ohio Utilities Protection Service 1-800-362-2764 Oil & Gas Producers Underground Protect'n Svc 1-800-925-0988
Connecticut Call Before You Dig 1-800-922-4455	Massachusetts Dig Safe System, Inc. 1-888-344-7233	Oklahoma Call Okie 1-800-522-6543
Delaware Miss Utility of Delmarva 1-800-282-8555	Michigan Miss Dig System, Inc. 1-800-482-7171	Oregon Oregon Utility Notification Center/One Call Concepts 1-800-332-2344
Florida Sunshine State One-Call of Florida, Inc. 1-800-432-4770	Minnesota Gopher State One Call 1-800-252-1168	Pennsylvania Pennsylvania One Call System, Inc. 1-800-242-1776
Georgia Underground Protection Center, Inc. 1-800-282-7411	Mississippi Mississippi One-Call System, Inc. 1-800-227-6477	Rhode Island Dig Safe System, Inc. 1-888-344-7233
Hawaii Underground Service Alert North 1-800-227-2600	Missouri Missouri One-Call System, Inc. 1-800-344-7483	South Carolina Palmetto Utility Protection Service Inc. 1-888-721-7877
Idaho Dig Line Inc. 1-800-342-1585 Kootenai County One-Call 1-800-428-4950 Shoshone - Benewah One-Call 1-800-398-3285	Montana Utilities Underground Protection Center 1-800-424-5555 Montana One Call Center 1-800-551-8344	South Dakota South Dakota One Call 1-800-781-7474
Illinois JULIE, Inc. 1-800-892-0123 Digger (Chicago Utility Alert Network) 312-744-7000	Nebraska Diggers Hotline of Nebraska 1-800-331-5666	Tennessee Tennessee One-Call System, Inc. 1-800-351-1111
Indiana Indiana Underground Plant Protection Service 1-800-382-5544	Nevada Underground Service Alert North 1-800-227-2600	
	New Hampshire Dig Safe System, Inc. 1-888-344-7233	

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ATTACHMENT 1 (Continued)

Texas

Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah

Blue Stakes of Utah
1-800-662-4111

Vermont

Dig Safe System, Inc.
1-888-344-7233

Virginia

Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington

Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating
Council
509-456-8000

West Virginia

Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin

Diggers Hotline, Inc.
1-800-242-8511

Wyoming

Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia

Miss Utility
1-800-257-7777

Alberta

Alberta One-Call Corporation
1-800-242-3447

British Columbia

BC One Call
1-800-474-6886

Ontario

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

Quebec

Info-Excavation
1-800-663-9228

Subject

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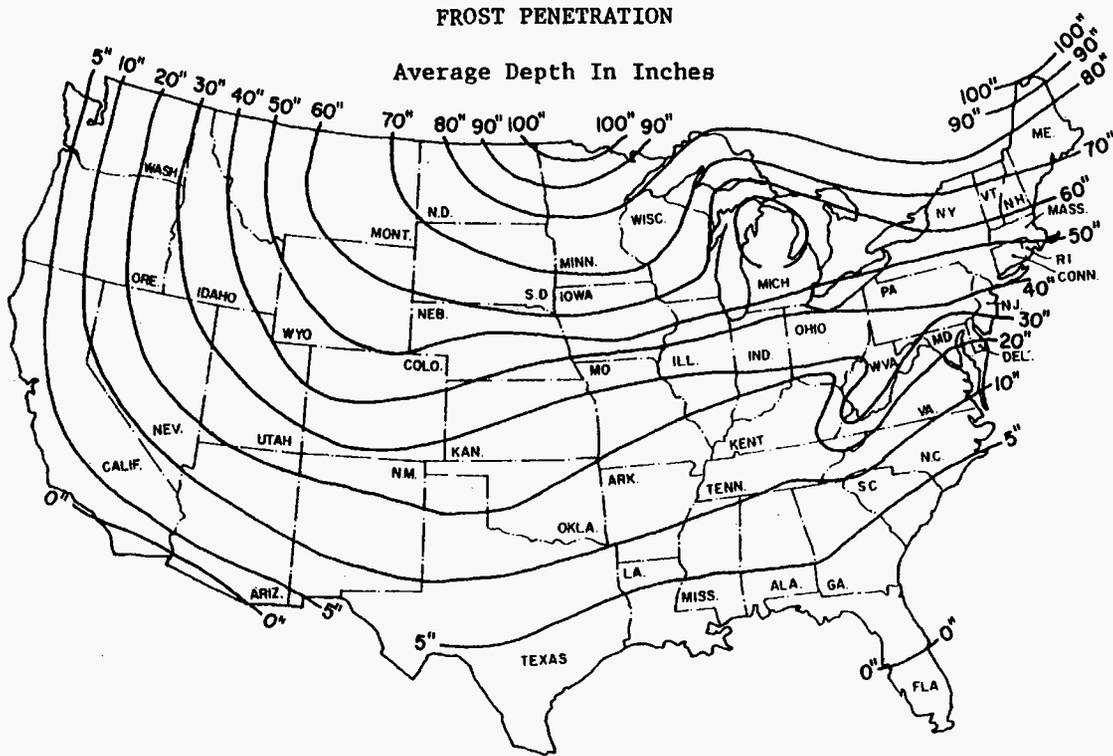
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ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

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**ATTACHMENT 3
UTILITY CLEARANCE FORM**

Client: _____ Project Name: _____
 Project No.: _____ Completed By: _____
 Location Name: _____ Work Date: _____
 Excavation Method/Overhead Equipment: _____

1. Underground Utilities Circle One
- a) Review of existing maps? yes no N/A
 - b) Interview local personnel? yes no N/A
 - c) Site visit and inspection? yes no N/A
 - d) Excavation areas marked in the field? yes no N/A
 - e) Utilities located in the field? yes no N/A
 - f) Located utilities marked/added to site maps? yes no N/A
 - g) Client contact notified yes no N/A
 Name _____ Telephone: _____ Date: _____
 - g) State One-Call agency called? yes no N/A
 Caller: _____
 Ticket Number: _____ Date: _____
 - h) Geophysical survey performed? yes no N/A
 Survey performed by: _____
 Method: _____ Date: _____
 - i) Hand excavation performed (with concurrent use of utility
 detection device)? yes no N/A
 Completed by: _____
 Total depth: _____ feet Date: _____
 - j) Trench/excavation probed? yes no N/A
 Probing completed by: _____
 Depth/frequency: _____ Date: _____

2. Overhead Utilities Present Absent
- a) Determination of nominal voltage yes no N/A
 - b) Marked on site maps yes no N/A
 - c) Necessary to lockout/insulate/re-route yes no N/A
 - d) Document procedures used to lockout/insulate/re-route yes no N/A
 - e) Minimum acceptable clearance (SOP Section 5.2): _____

3. Notes:

Approval:

 Site Manager/Field Operations Leader Date

c: PM/Project File
 Program File

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**ATTACHMENT 4
OSHA LETTER OF INTERPRETATION**

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

***Question:** Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

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ATTACHMENT 4 (Continued)

When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, “acceptable means” must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either “other acceptable means” or “safe and acceptable means.” The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified “careful probing or hand digging” as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language “to allow other, *equally effective means* of locating such installations.” The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used where the utilities have not been marked and detection equipment is not being used – “probing with hand-held tools.” This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

OSHA received two comments * * * and input from ACCSH [OSHA’s Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping ‘such as probing with hand-held tools’ from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of “acceptable means” in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a “shooter” (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an “acceptable means” for locating underground utilities.

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ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA's interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at <http://www.osha.gov>.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	SA-2.5	Page	1 of 6
Effective Date	09/03	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>DS</i>		

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

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Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.
- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.
- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.
- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.

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- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

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6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.
- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.
- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (± 10 percent), the well may be sampled.
- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.
- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).
- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

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**ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS**

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

- I. Work limited to the following (description, area, equipment used): Monitoring well drilling and installation through direct push technology
- II. Required Monitoring Instruments: _____
- III. Field Crew: _____
- IV. On-site Inspection conducted Yes No Initials of Inspector _____

TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

- | | | |
|--|--|--|
| V. Protective equipment required | Respiratory equipment required | |
| Level D <input checked="" type="checkbox"/> Level B <input type="checkbox"/> | Full face APR <input type="checkbox"/> | Escape Pack <input type="checkbox"/> |
| Level C <input type="checkbox"/> Level A <input type="checkbox"/> | Half face APR <input type="checkbox"/> | SCBA <input type="checkbox"/> |
| Detailed on Reverse | SKA-PAC SAR <input type="checkbox"/> | Bottle Trailer <input type="checkbox"/> |
| | Skid Rig <input type="checkbox"/> | None <input checked="" type="checkbox"/> |

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

- | | | |
|--------------------------|-----------------|-------------------|
| VI. Chemicals of Concern | Action Level(s) | Response Measures |
| _____ | _____ | _____ |

VII. Additional Safety Equipment/Procedures

- | | |
|---|--|
| Hard-hat <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Hearing Protection (Plugs/Muffs) <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
| Safety Glasses <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Safety belt/harness <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Chemical/splash goggles <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No | Radio <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Splash Shield <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No | Barricades <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
| Splash suits/coveralls <input type="checkbox"/> Yes <input type="checkbox"/> No | Gloves (Type - _____) <input type="checkbox"/> Yes <input type="checkbox"/> No |
| Steel toe Work shoes or boots <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Work/warming regimen <input type="checkbox"/> Yes <input type="checkbox"/> No |

Modifications/Exceptions: Reflective vests for high traffic areas.

- | | | | | |
|--|-------------------------------------|-------------------------------------|--------------------------|--------------------------|
| VIII. Procedure review with permit acceptors | Yes | NA | Yes | NA |
| Safety shower/eyewash (Location & Use)..... | <input type="checkbox"/> | <input checked="" type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| Daily tail gate meetings..... | <input checked="" type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| Contractor tools/equipment/PPE inspected | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

IX. Site Preparation

- | | | |
|---|------------------------------|-----------------------------|
| Utility Clearances obtained for areas of subsurface investigation | <input type="checkbox"/> Yes | <input type="checkbox"/> No |
| Physical hazards removed or blockaded | <input type="checkbox"/> Yes | <input type="checkbox"/> No |
| Site control boundaries demarcated/signage | <input type="checkbox"/> Yes | <input type="checkbox"/> No |

X. Equipment Preparation

- | | | |
|---|------------------------------|--|
| Equipment drained/depressurized | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Equipment purged/cleaned | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Isolation checklist completed | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Electrical lockout required/field switch tested | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Blinds/misalignments/blocks & bleeds in place | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Hazardous materials on walls/behind liners considered | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |

- XI. Additional Permits required (Hot work, confined space entry)..... Yes No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____



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

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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

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 15 of 16
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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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Effective Date 01/28/2009	Revision 6
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

Attachment A iDW Label

INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE _____ JOB NO. _____

LOCATION _____

DATE _____

DRUM# _____

CONTENTS _____

VOLUME _____

CONTACT _____

EMERGENCY PHONE NUMBER _____

APPENDIX C

**ANALYTICAL LABORATORIES OF FLORIDA, INC.
STANDARD OPERATING PROCEDURES AND CERTIFICATION**

ANALYTICAL LABORATORIES OF FLORIDA, INC.
STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: ANALYSIS OF SELECTED VOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY USING A MASS SPECTROPHOTOMETER
IN WATER AND SOIL MATRICES

DOCUMENT CONTROL NUMBER: ALF09-001-Revision 0

EFFECTIVE DATE: 4/21/09

APPROVALS:

MANAGER: _____ Date: _____

QA MANAGER: _____ Date: _____

LAB DIRECTOR: _____ Date: _____

ANALYTICAL LABORATORIES
Analytical Laboratories of Florida
Standard Operating Procedure

Method 8260

Aromatic and Halogenated Volatiles by Gas Chromatography/Mass Spectrometry

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to analyze volatile organic compounds in ground water and soils.

1.2 The following compounds are currently determined using method 8260:

Dichlorodifluoromethane	1,1,2-Trichloroethane	Chloromethane
Dibromochloromethane	Vinyl Chloride	Chlorobenzene
Bromomethane	Ethylbenzene	Chloroethane
m,p-Xylenes	Trichlorofluoromethane	o-Xylene
1,1-Dichloroethene	Bromoform	Methylene Chloride
1,1,2,2-Tetrachloroethane	t-1,2-Dichloroethene	1,3-Dichlorobenzene
MTBE	1,4-Dichlorobenzene	1,1-Dichloroethane
1,2-Dichlorobenzene	Chloroform	Naphthalene
Carbon Tetrachloride	cis-1,2-Dichloroethene	1,1,1-Trichloroethane
2,2-Dichloropropane	Benzene	Bromochloromethane
1,2-Dichloroethane	Trichloroethene	1,3-Dichloropropane
1,2-Dichloropropane	Bromodichloromethane	Styrene
c-1,3-Dichloropropene	1,1,1,2-Tetrachloroethane	Toluene
Isopropylbenzene	Tetrachloroethene	Bromobenzene
t-1,3-Dichloropropene	n-Propylbenzene	2-Chlorotoluene
1,3,5-Trimethylbenzene	tert-Butylbenzene	Acrolein
sec-Butylbenzene	p-Isopropyltoluene	n-Butylbenzene
1,2,4-Trimethylbenzene	Ethanol	Acetone
Acrylonitrile	Freon 113	Acetonitrile
Allyl chloride	Carbon disulfide	Propionitrile
Vinyl acetate	2-Butanone (MEK)	Methacrylonitrile
Isobutyl alcohol	1,1-Dichloropropene	Dibromomethane
1,2,3-Trichlorobenzene	Methyl methacrylate	2-Hexanone
Ethyl methacrylate	1,2-Dibromoethane	4-Chlorotoluene
t-1,4-Dichloro-2-butanone	1,2,3-Trichloropropane	DBCP
c-1,4-Dichloro-2-butanone	Pentachloroethane	Hexachlorobutadiene
1,2,4-Trichlorobenzene	1-methylnaphthalene	Iodomethane
2-Chloro-ethyl-vinylether	2-methylnaphthalene	1,4-Dioxane
4-Methyl-2-Pentanone		

2.0 SUMMARY OF METHOD

2.1 Method 8260 is used to determine volatile organic compounds in samples by Purge-and-Trap (Method 5030/5035). The analytes are introduced directly onto a narrow-bore column that is directly interfaced to the ion source. The compounds are identified by retention times and comparing the mass spectra with the spectra of standards. The quantitative results are determined by comparing the response of a quantitative ion to an internal standard and using a minimum of a five-point calibration curve.

3.0 INTERFERENCE

3.1 Sample pathways can become contaminated if the pathway is exposed to samples with high levels of target analytes present. In the case where an analyte is present at a concentration of over 200ppb, samples following this should not be reported for this analyte until a blank or sample is analyzed and found to be below lowest quantitative value reported or "clean."

4.0 DEFINITIONS

CCV- Continuing Calibration Verification- is a procedure to determine whether an instrument was within acceptable calibration throughout the period in which samples were analyzed (i.e., to verify that the initial calibration was applicable during the sample analyses).

COC- Chain of Custody- a record that documents the possession of the samples from the time of collection to receipt in the lab. This record generally includes the number and types of samples, the sampler, the collection time and date, and the requested analyses

CVS- Calibration Verification Standard- An intermediate concentration level standard prepared, analyzed and evaluated against the initial calibration curve. This standard is analyzed every 12 hours and must meet the acceptance criteria as noted in EPA Method 8260 Section 7.4.4 through 7.4.7

DOC- Demonstration of Capability- the procedure to establish the ability of an analyst to generate acceptable accuracy. May be Initial (IDOC) or Continuing (CDOC) procedure..

DOH- Department of Health

DI – De-ionized Water (may substitute any other contaminant free water)

GC/MS- Gas Chromatograph/Mass Spectrometer

IC- Initial Calibration as indicated in Section 9 of this SOP

ICB- Initial Calibration Blank- Blank analyzed following an initial calibration.

ICV- Initial Calibration Verification- similar to CVS but analyzed immediately following an initial calibration.

LCS - Laboratory Control Sample- A sample matrix free from the analytes of interest spiked with known concentrations of analytes of interest. Used to establish intra-laboratory precision and to assess the performance of the measurement system.

LOD- Limit of Detection. Equivalent to MDL.

LOQ- Limit of Quantitation. Equivalent to PQL.

MB- Method Blank. Equivalent to RB and Blank. A sample of a matrix similar to the batch of associated samples that is intended to contain none of the analytes of interest and which is processed with and under the same conditions as the associated samples

MDL – Method Detection Limit- Equivalent to LOD. MDL is the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero, and is determined from analysis of a sample in a given matrix containing the analyte.

MeOH – Purge and Trap Grade Methanol

MS- Matrix Spike- A selected sample from the analytical set spiked with the appropriate test specific standard and surrogate.

MSD- Matrix Spike Duplicate- A second or duplicate selected sample from the analytical set spiked with the appropriate test specific standard and surrogate.

NA- Not Applicable

NELAC- National Environmental Laboratory Accreditation Conference- A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

NELAP- National Environmental Laboratory Accreditation Program- The overall program of which NELAC is a part.

ND- Not Detected - The value is less than the MDL.

PPB- parts per billion

PQL – Practical Quantitation Limit- Equivalent to LOQ. PQL is a quantitation limit that represents a practical and routinely achievable quantitation limit with a high degree of certainty (>99.9% confidence) in the results.

QA- Quality Assurance- An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets the defined standards of quality with a stated level of confidence.

QC- Quality Control- The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the need of users.

RB- Reagent Blank. Equivalent to MB and Blank

RL – Reporting Limit

RPD- Relative Percent Difference- the difference between the amount measured and the true value, expressed as a percentage

SOP- Standard Operating Procedure- A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method of performing certain routine or repetitive tasks.

Standard Deviation- A computed measure of variability indicating the spread of the data set around the mean.

VOA- Volatile Organic Analysis or Volatile Organic Aromatics

VOC- Volatile Organic Compounds

VOH- Volatile Organic Halocarbons

5.0 METHOD DETECTION LIMITS

- 5.1 The method detection limit is defined as the minimum concentration of substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.
- 5.2 MDL's should be determined using the procedure as described in 40CFR Part 136 Appendix B. Prepare and analyze 7 replicate LCS at the concentration required to achieve necessary MDL's. Calculate the Standard Deviation and the Method Detection Limit using the Student "T" factor of 3.143 for 7 replicates.
- 5.3 The MDL's for this method are included in the tables 18.4 and 18.5 below.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Teledyne Purge and Trap Concentrator – Stratum with 25 ml sparge tube
- 6.2 GC/MS: HP5975C VL-MDS Triple Axis Detection with HP 7890A GC- J&W DB-VRX column 20meter, 0.18mm ID, 1um df)
- 6.3 Syringes- Hamilton Gas Tight, 1ul-10ml
- 6.4 Class A Volumetric Flasks, 50 ml
- 6.5 Balance- Acculab ProPocket-250B

7.0 REAGENTS AND STANDARDS

- 7.1 Organic Free Water
- 7.2 Methanol (MeOH), CH₃OH- Purge and Trap Grade
- 7.3 Stock Standards-Purchased as Certified Solutions

8.0 SAMPLE COLLECTION, SHIPMENT, HANDLING AND PRESERVATION

- 8.1 Water Samples-The sampling process should yield a minimum of 2 vials (20-40mls each), filled to the top, with no air bubbles present. The samples must be kept on ice until they have reached the lab and have been logged in. Once the samples have been logged in, they are kept in a refrigerator at 4 degrees Celsius. If the samples were not preserved during sampling the hold time for those samples is 7 days. If the samples were preserved (with Hydrochloric acid) the hold time is 14 days.
- 8.2 Soil Samples- The sampling process should yield 1 soil vial per sample. . The samples must be kept on ice until they have reached the lab and have been logged in. Once the samples have been logged in, they are kept in a refrigerator at 4 degrees Celsius. Samples hold time for those samples is 14 days.

9.0 CALIBRATION, STANDARDIZATION, PROCEDURES and CALCULATIONS

9.1 Tuning- Prior to analysis of samples or standards the Mass Spec must pass a BFB tune. To start the process, an Autotune is performed through the **Instrument Control** screen (**GC/MS Instrument #1**). Choose the options below to Autotune the MS.

1. **View**
2. **Manual Tune**
3. **Tune**
4. **Autotune**

The software will automatically start the MS tune. After completion of the tune, a report will be generated, and the results can be accepted according to the following criteria listed in the GC/MS software:

>> The mass assignments shown in the upper “profile” part of the display should be within +/- 0.2 amu of 69, 219, and 502.

>> The peak widths (PW) of these three peaks should be 0.5 +/- 0.1 amu.

>> The mass assignments shown in the lower “scan” part of the display should be within +/- 0.1 amu of 69, 219, and 502.

>> The relative abundance should show that the peak at 69 amu is the largest.

>> The Isotope mass assignments should each be 1 amu greater than the mass assignments of the parent peaks.

>> The Isotope ratio figures should be close to the theoretical values of 1.08 for m/z 69, 4.32 for m/z 219, and 10.09 for m/z 502.

>> If mass 28 is greater than mass 18, there may be an air leak in the system. Exceptions are when it is within 1 hour of venting or during the first autotune after refilling the calibration vial.

Go to **File** and **Save Tune Values** to file **BFB1**. To return to the Instrument Controls choose **View** and then **Instrument Controls**.

Once the Autotune has passed, analyze a blank spiked with BFB at a concentration between 5-50ng. When BFB is chosen as the sample type in the sequence (at **MS Top**) the report will automatically print out and each parameter will be listed with a Pass or Fail. Once the BFB tune passes analysis can begin. In addition, a BFB tune should be performed every 12 hours while samples are being analyzed to ensure that all of the MS parameters met specified mass intensity criteria. As long as the BFB tune continues to pass every 12 hours, the tuning requirements have

been fulfilled. If the BFB tune fails, perform an Autotune and /or adjust MS parameters until a BFB tune passes. If the BFB tune continues to fail, it may be a sign that there is an instrument problem such as a dirty ion source. See Table 18.3 for Acceptance Criteria.

- 9.2 Quantitation Database-The Quantitation Database stores information about each compound including Retention Times, Target Ions, Curve Fit, Compound Attributes, and Calibration Levels. Go to the **Environmental Data Analysis** screen and under **Initial Cal** there is an option to **Set Up Quantitation**. Using a data file where all compounds are present, identify each compound and insert it into the Quantitation Database starting with the first internal standard. Second, enter the compound with the shortest retention time until all the compounds of interest are listed. After all of the compounds have been identified and entered into the database, additional information can be inserted as well. For example, Method 8260 recommends that certain compounds have specific functions or attributes (page 2 of the database). They are as follows:

Surrogate Standards- toluene-d8, 4-bromofluorobenzene,
1,2-dichloroethane-d4, and dibromofluoromethane

Internal Standards- fluorobenzene, chlorobenzene-d5, and
1,4-dichlorobenzene-d4

Tune Standard- 4-bromofluorobenzene

System Performance Check Compounds- chloromethane,
1,1-dichloroethane, bromoform,
chlorobenzene, 1,1,2,2-tetrachloroethane

Calibration Check Compounds- 1,1-dichloroethene, chloroform,
1,2-dichloropropane, toluene,
ethylbenzene, vinyl chloride

- 9.3 Water Calibration-Once the Quantitation Database has been prepared, a calibration curve can be analyzed and the levels updated. Make nine levels of standards that contain all of the compounds that are required for 8260 analysis. Note that all working standards are prepared in 50ml of deionized water and immediately transferred to a 40ml volatile vial for analysis.
- 9.3.1 Analyze the 9 working standards. Now there is sufficient information to plot the response of the compounds at different concentration levels. Go to the **Environmental Data Analysis** screen, open the file that corresponds to the first standard (ex: level 7, 100ppb) by selecting **File/ Load Data File**. Once the file is opened, select **InitCal/Update Levels**. Select **Recalibrate** and select **7** from the calibration level ID selection box. **Replace** the Responses and the Retention Times, and select **Do Update**. Repeat this step until each level is entered.

- 9.3.2 After all of the levels are entered, go to **InitCal/Edit Compounds** choose a compound and select **View**. On **Page 3**, all of the levels should be listed with the concentration, and response. Select **Plot** and view the Relative Standard Deviation for the Response Factor. An acceptable RF RSD is less than 20% with the exception of any compound specified to be a Calibration Check Compound (acceptable limits are less than 30%). View each compound, and make sure that the criterion for the RF has been met. Up to two points may be removed from the curve to bring the RF RSD into range. If the lowest level is removed from a compound, the detection limit will change, and if the highest point is removed the reporting limit will change. If an acceptable RF RSD can not be obtained for one or more compounds, with a 5-point calibration, Linear Regression may be used to determine if the calibration is acceptable for that compound. In addition, if the mean of the RSD value for all the compounds in the calibration is less than 20% the calibration is acceptable. The Response Factor is calculated as follows:

$$\text{RF} = \frac{\text{As} \times \text{Cis}}{\text{Ais} \times \text{Cs}}$$

Where:

As = Peak Area of the analyte or surrogate

Ais = Peak Area of the Internal Standard

Cs = Concentration of the analyte or surrogate

Cis = Concentration of the Internal Standard

- 9.3.3 Save the method after all necessary changes have been made. Go to **File/Save Method** and select **OK**.
- 9.4 Soil Calibration-Once the Quantitative Database has been prepared, a calibration curve can be analyzed and the levels updated. Make nine levels of standards that contain all of the compounds that are required for 8260 analysis. For help making standards, see Section 1- Making Standards. Note that all working standards are prepared in 10 ml of analyte – free water.
- 9.4.1 Next, analyze the 9 working standards by following the instructions in the SOP **Method 5035-Purge-and-Trap Analysis for Soil Samples**. Now there is sufficient information to plot the response of the compounds at different concentration levels. Go to the **Environmental Data Analysis** screen, open the file that corresponds to the first standard (level 7, 100ppb) by selecting **File/ Load Data File**. Once the file is opened, select **InitCal/Update Levels**. Select **Recalibrate** and select **7** from the calibration level ID selection box. **Replace** the Responses and the

Retention Times, and select **Do Update**. Repeat this step until each level is entered.

- 9.4.2 After all of the levels are entered, go to **InitCal/Edit Compounds** choose a compound and select **View**. On **Page 3** all of the levels should be listed with the concentration, and response. Select **Plot** and view the Relative Standard Deviation for the Response Factor. An acceptable RF RSD is less than 20% with the exception of any compound specified to be a Calibration Check Compound (acceptable limits are less than 30%). View each compound, and make sure that the criterion for the RF has been met. Up to two points may be removed from the curve to bring the RF RSD into range. If the lowest level is removed from a compound, the detection limit will change, and if the highest point is removed the reporting limit will change. If an acceptable RF RSD can not be obtained for one or more compounds, with a 5-point calibration, Linear Regression may be used to determine if the calibration is acceptable for that compound. In addition, if the mean of the RSD value for all the compounds in the calibration is less than 20% the calibration is acceptable. The Response Factor is calculated as follows:

$$\text{RF} = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Peak Area of the analyte or surrogate

A_{is} = Peak Area of the Internal Standard

C_s = Concentration of the analyte or surrogate

C_{is} = Concentration of the Internal Standard

- 9.4.3 Save the method after all necessary changes have been made. Go to **File/Save Method** and select **OK**.
- 9.5 Sample Analysis- Samples are analyzed in batches. A batch consists of a Method Blank, a Laboratory Control Standard, a Matrix Spike, Matrix Spike Duplicate, and up to 20 samples. Batches must be specific to the sample matrix; waters and soils can not be in the same batch.
- 9.5.1 GC/MS calibration verification- Consists of 3 steps that are performed at the beginning of each 12-hour analytical shift.
- 9.5.1.1 Prior to the analysis of samples or calibration standards, introduce 5-50ng of 4-bromofluorobenzene (BFB) standard into the GCMS. The resultant mass spectra for the BFB must meet the criteria in Table 18.3 before sample analysis begins. This criteria must be

demonstrated each 12-hour shift during which samples are analyzed.

9.5.1.2 The initial calibration curve for each compound of interest should be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing a CCV. The results should meet the verification acceptance criteria provided. Note: The BFB and CCV may be combined into a single standard as long as both acceptances criteria can be met with out interference.

9.5.1.3 A method blank should be analyzed during the calibration verification procedure or at any other time during the analytical shift to ensure that the total system is free of contaminants. If the method blank indicates contamination, reanalyze in the event the contamination is carry over from a previous sample. See Section 8.0 of Method 8000 for method blank performance criteria.

9.5.2 Set up samples in the desired order of analysis in the Teledyne Stratum Purge and Trap Concentrator. Set up the method to be analyzed on the concentrator using the front control panel. Then create a sequence as listed below so that the GC software will collect data from the Mass Spec.

9.5.3 To run a sequence go to the **GC/MS Instrument #1 MS Top/Environmental** screen.

1. Select **Method**, then **Load**.
2. The current method being used is **8260.M**
3. **OK**
4. Select **Sequence/Edit Sample Log Table**
5. Fill in the Log Table.
6. Select **OK**
7. Select **Save**
8. Save the file as **DDMMYY.S**
9. Select **OK, Run, and Run Sequence**.

9.6 Acquiring Data-As the GC/MS is running a sample, it can be viewed through the Environmental Data Analysis screen. Go to File/Take Snapshot. This option can only be used when a sample run is in progress; once the sample run is completed the file can only be viewed by File/ Load Data File. Quantitation can take place with both of these options. Select Quant/ Calculate/ Generate Report. Then the option will be given to show the results on the screen and/or print the results. Once the options have been chosen, select OK. At this point, the data should be collected and reviewed for reporting.

9.7 Calculations

Dry weight determination:

$$\text{mg/dry kg PH} = \frac{C_s}{1 - (\% \text{moisture}/100)}$$

Where: C_s = Concentration of Petroleum Hydrocarbons (mg/L or mg/kg)

Matrix Spike Recovery

$$\% \text{ Recovery} = [(A - B) / C] \times 100$$

Where: A = measured concentration of spiked sample

B = measured concentration of sample

C = actual spike concentration

Duplicate Precision

$$\text{RPD} = \frac{A}{B} \times 100$$

Where: A = Absolute value of Dup 1 – Dup 2

B = average of Dup 1 and Dup 2

LCS Recovery

$$\% \text{ Recovery} = (A / B) \times 100$$

Where: A = measured concentration of spiked sample

B = actual concentration of sample

Surrogate Recovery

$$\% \text{ Recovery} = (A / B) \times 100$$

Where: A = concentration found

B = concentration added

10.0 DATA ASSESSMENT and ACCEPTANCE CRITERIA

- 10.1 Reviewing Data-All the data should be reviewed thoroughly to ensure that it is as accurate as possible. To begin the Method Blank must be “clean” or below the reporting limit. The LCS and MS/MSD should have a percent recovery +/- 30%. The samples Internal Standard recovery must be between 50% and 200% of the Internal Standard recovery in the Laboratory Control Standard. Once these Quality Control parameters have been met, the data can be reviewed in closer detail to determine if the results are reportable or if the sample should be reanalyzed. Begin by using the CVS Quantitation Report as a reference to compare sample Quantitation Reports. As stated above, make sure that the Internal Standard recoveries are within QC limits (50%-200%). In addition, it is important to verify that the Retention Times of each analyte are within 0.1

minutes of the CVS Retention Time for that analyte. The Qvalue should also be used to determine if the result of a particular compound is reportable. Currently, the analyte results are considered reportable if the Qvalue is over 80, but exceptions are made if there appears to be matrix interference and/or the chromatogram is examined manually. Below is the definition of the Qvalue as described by the **HELP** section of the software:

The Qvalue is an approximate measure of the similarity between the expected intensities of the quantitation and qualifier ions and the actual intensities. The Qvalue should not be construed as an absolute measure of the quality of the closeness of fit. The Qvalue only examines the intensities of the quantitation ion and the qualifying ions. When a compound has no qualifier ions, the Qvalue will always be 100.

- 10.2 Next, examine the concentration of each analyte. The concentration (in ppb) is not reportable below the Method Detection Limit and over 20% above the highest point on the calibration curve. If any analyte in a sample is outside any of these parameters, the sample should be reanalyzed (possibly at a different dilution) to achieve reportable results. Samples were analytes greater than 20% above the range of calibration may be reported if the proper qualifiers are used.

- 10.3 All results should be recorded on the GC/MS Volatiles Bench sheet or on the original chromatograph. These results may be used in reporting or as a reference depending on whether or not the sample met the appropriate QC requirements (listed above).

- 10.4 Reporting Data- Once all of the results for a specific work order are complete, the results can be reported on an Excel spreadsheet. Templates for the Excel spreadsheets are saved in Windows Explorer on the C drive. To locate a template, go to Start, Programs, Windows Explorer, and select the method to be reported (ex: 8021b Water). Use the information provided in the Log In sheet and the GC Bench sheet to fill in the Excel template. As the results are being transferred from the GC Bench sheet to the Excel spreadsheet, make sure that any analyte results above the MDL (currently 1 ppb) are highlighted using the Bold option and reported using two significant digits. When the template has been filled out completely, go to File, Save As, and use the Work Order number to save the template as a results file.

Please reference NELAC Quality Systems Chapter 5 Appendix C and D for additional guidance of acceptance criteria.

11.0 QUALITY CONTROL

- 11.1 Refer to Chapter One and Method 8000 for specific quality control procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.
- 11.2 Quality control (QC) procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:
- 11.2.1 The GC/MS system must be tuned to meet the BFB specifications. In Secs. 7.3.1 and 7.4.1 (See Tables, diagrams,... of this SOP for passing criteria)
 - 11.2.2 There must be an initial calibration of the GC/MS system as described in Sec 7.3.
 - 11.2.3 The GC/MS system must meet the SPCC criteria in Sec 7.4.4 and CCC criteria in Sec 7.4.5 each 12 hours of analysis.
- 11.3 Initial Demonstration of Proficiency – Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec 8.0 for information on how to accomplish this demonstration.
- 11.3.1 Acceptance Criteria – For the Initial and Continuing Demonstration of Capability to be acceptable, the mean recovery for all compounds should be between 80 – 120% and the Standard Deviation less than 20%.
- 11.4 Sample Quality Control for Preparation and Analysis – The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.
- 11.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

- 11.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.
- 11.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
- 11.4.4 See Method 8000, Sec 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.
- 11.5 Surrogate recoveries – The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.
- 11.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the Calibration Verification Standard (CVS) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is performing acceptably, the injector is leaking, the injection septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.
- 11.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.
- 11.8 Biannual LCS Check- As indicated in NELAC Quality Systems Chapter 5 appendix D. For test methods that have extremely long lists of analytes, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period. Acceptance criteria same as LCS

- 11.9 Limit of Detection- The laboratory shall determine the LOD for the method for each target analyte of concern in the quality system matrices. Acceptance criteria same as LCS
- 11.10 Limit of Quantitation- The laboratory shall determine the LOQ for each analyte of concern according to a defined documented procedure. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. Acceptance criteria same as LCS.

For additional assistance, please reference NELAC Quality System Chapter 5 Appendix C.

- 11.11 Proficiency Testing- Proficiency testing is performed in accordance to NELAC requirements. The results are also used as intra-laboratory examples if continuing demonstration of performance.

12.0 METHOD PERFORMANCE

- 12.1 This method has been tested using purge –and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore column, water was spiked at concentrations between 0.5 and 10 ug/l. Single laboratory accuracy and precision data are presented for the method analytes in the tables.
- 12.2 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand, a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon, and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 4ug/kg. These data is listed in the tables. All data were calculated using fluorobenzene as the internal standard added to the sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.
- 12.2.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.
- 12.2.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in the tables include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylene and trimethylbenzenes. This is due to contamination of the soil prior to sample collection and to the fact that no background was subtracted.

13.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Corrective actions for out-of-control or unacceptable data may include consultation with the QA Officer and/or the Lab Director. No list can anticipate every possible corrective action. The following is a list of the most commonly preformed corrective actions. Ultimately, consultation with the client is recommended if these corrective actions fail to produce satisfactory results.

13.1.1 Sample reanalysis

13.1.2 Sample re-extraction

13.1.3 Instrument Maintenance

13.1.4 Evaluation of calibration curve, including continuing calibration verification and/or initial calibration.

13.1.5 Notification of client

14.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL DATA

14.1 As noted in 13.0, the ultimate contingency for handling out-of-control or unacceptable data is consultation with the client. There are situations with some sample matrices which will not produce data with the confidence that is achievable with other samples. When corrective actions fail to correct a problem with unacceptable or out-of-control data, the results shall be discussed with the client by the QA Officer and/or the Lab Director.

15.0 SAFETY

15.1 The toxicity or 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. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. All personnel should wear gloves, a lab coat, safety goggles, long pants, and completely encasing shoes to help ensure safety.

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 All laboratories generate wastes. Some wastes can be hazardous such as acidic wastes, metal bearing wastes and organic wastes.

16.1.2 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

16.1.3 Non-Hazardous waste with other pH levels may be directly poured in the sink.

16.1.4 Refer to SOP S.2, Disposal of Samples and SOP S.13, Sample Storage and Disposal , for further information.

17.0 REFERENCES

1. Method 8260-Volatile Organic Compounds By Gas Chromatography/ Mass Spectrometry (GC/MS), U.S. Environmental Protection Agency, Revision 2, December 1996.
2. Method 8000-Determinative Chromatographic Separations, U.S. Environmental Protection Agency, Revision 3, December 1996.
3. Method 5030B-Purge and Trap for Aqueous Samples, U.S. Environmental Protection Agency, Revision 2, December 1996.
4. Method 5035-Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, U.S. Environmental Protection Agency, Revision 0, December 1996.
5. Analysis of Selected Halogenated and Aromatic Volatile Organic Compounds by GC in Water and Soil Matrices. Document 04001R0, Analytical Laboratories of Florida April 2004.
6. NELAC Quality Manual for Analytical Laboratories of Florida, Revision 2, November 2006.

18.0 TABLES, DIAGRAMS FLOWCHARTS, VALIDATION DATA

18.1 Water Calibration Curve Preparation and Concentration made in 50ml volumetric flask

Level	Conc.	Cal Std	Water Vol.	IS added	Amount Present	GC/MS Description
Level 9	200 ppb	200ul	50 ml	5.0 ul	200 ppb	ICV 9
Level 8	150 ppb	150ul	50 ml	5.0 ul	150 ppb	ICV 8
Level 7	100ppb	100ul	50 ml	5.0 ul	100 ppb	ICV 7
Level 6	80ppb	80ul	50 ml	5.0 ul	80 ppb	ICV 6
Level 5	60ppb	60ul	50 ml	5.0 ul	60 ppb	ICV 5
Level 4	40ppb	40ul	50 ml	5.0 ul	40 ppb	ICV 4
Level 3	20ppb	20ul	50 ml	5.0 ul	20 ppb	ICV 3
Level 2	5ppb	5ul	50 ml	5.0 ul	5 ppb	ICV 2
Level 1	1ppb	1ul	50 ml	5.0 ul	1 ppb	ICV 2
Level CCV	20ppb	100ul	50 ml	5.0 ul	100 ppb	CCV1

18.2 Soil Calibration Curve Preparation and Concentration made in 22ml soil vials

Soil Curve			Cal Std	Soil Vol.	IS added
Level 9	200 ppb	10 ml H2O + 5gr CS	20ul	5ml	5ul
Level 8	150 ppb	10 ml H2O + 5gr CS	15ul	5ml	5ul
Level 7	100ppb	10 ml H2O + 5gr CS	10ul	5ml	5ul
Level 6	80ppb	10 ml H2O + 5gr CS	8ul	5ml	5ul
Level 5	60ppb	10 ml H2O + 5gr CS	6ul	5ml	5ul
Level 4	40ppb	10 ml H2O + 5gr CS	4ul	5ml	5ul
Level 3	20ppb	10 ml H2O + 5gr CS	2ul	5ml	5ul
Level 2	10ppb	10 ml H2O + 5gr CS	1ul	5ml	5ul
Level 1	5ppb	10 ml H2O + 5gr CS	.5ul	5ml	5ul
Level CCV	100ppb	10 ml H2O + 5gr CS	10ul	5ml	5ul

18.3 BFB Tuning Acceptance Criteria

Target Mass	Relative to Mass	Lower - Upper Acceptance %
50	95	8 - 40
75	95	30 - 60
95	95	100 - 100
96	95	5 - 9
173	174	0.00 - 2
174	95	50 - 200
175	174	5 - 9
176	174	93 - 101
177	176	5 - 9

1,2-Dichloropropane
Trichloroethene
Bromodichloromethane
1,4-Dioxane
Methyl methacrylate
2-chloro-ethyl-vinylether
c-1,3-Dichloropropene
4-methyl-2-pentanone
t-1,3-Dichloropropene
1,1,2-Trichloroethane
Toluene
1,3-Dichloropropane
Dibromochloromethane
Ethyl methacrylate
2-Hexanone
1,2-Dibromoethane
Tetrachloroethene
1,1,1,2-
Tetrachloroethane
Chlorobenzene
Ethylbenzene
Bromoform
m/p-Xylene
t-1,4-Dichloro-2-
butanone
Styrene
1,1,2,2-
Tetrachloroethane
o-Xylene
1,2,3-Trichloropropane
c-1,4-Dichloro-2-
butanone
Isopropylbenzene
Bromobenzene
n-Propylbenzene
2-Chlorotoluene
4-Chlorotoluene
Pentachloroethane
1,3,5-Trimethylbenzene
Tert-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
p-Isopropyltoluene
1,2-Dichlorobenzene
n-butylbenzene
DBCP
1,2,4-Trichlorobenzene
Naphthalene

1,2-Dichloropropane
Trichloroethene
Bromodichloromethane
1,4-Dioxane
Methyl methacrylate
2-chloro-ethyl-vinylether
c-1,3-Dichloropropene
4-methyl-2-pentanone
t-1,3-Dichloropropene
1,1,2-Trichloroethane
Toluene
1,3-Dichloropropane
Dibromochloromethane
Ethyl methacrylate
2-Hexanone
1,2-Dibromoethane
Tetrachloroethene
1,1,1,2-
Tetrachloroethane
Chlorobenzene
Ethylbenzene
Bromoform
m/p-Xylene
t-1,4-Dichloro-2-
butanone
Styrene
1,1,2,2-
Tetrachloroethane
o-Xylene
1,2,3-Trichloropropane
c-1,4-Dichloro-2-
butanone
Isopropylbenzene
Bromobenzene
n-Propylbenzene
2-Chlorotoluene
4-Chlorotoluene
Pentachloroethane
1,3,5-Trimethylbenzene
Tert-Butylbenzene
1,2,4-Trimethylbenzene
sec-Butylbenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
p-Isopropyltoluene
1,2-Dichlorobenzene
n-butylbenzene
DBCP
1,2,4-Trichlorobenzene
Naphthalene

Hexachlorobutadiene
1,2,3-Trichlorobenzene
2-Methylnaphthalene
1-Methylnaphthalene

ANALYTICAL LABORATORIES OF FLORIDA, INC.
STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: ANALYSIS OF SELECTED VOLATILE ORGANIC COMPOUNDS
BY GAS CHROMATOGRAPHY USING A MASS SPECTROPHOTOMETER
SIM MODE (EPA 8260 SIMS) IN WATER AND SOIL MATRICES

DOCUMENT CONTROL NUMBER: ALF11-001-Revision 0

EFFECTIVE DATE: 2/18/11

APPROVALS:

MANAGER: _____ Date: _____

QA MANAGER: _____ Date: _____

LAB DIRECTOR: _____ Date: _____

ANALYTICAL LABORATORIES
Analytical Laboratories of Florida
Standard Operating Procedure

Method 8260 SIM Mode
Aromatic and Halogenated Volatiles by Gas Chromatography/Mass Spectrometry

1.0 SCOPE AND APPLICATION

- 1.1 Method 8260 is used to analyze volatile organic compounds in ground water and soils.
- 1.2 The following compound(s) are currently determined using method 8260SIM:

1,4-Dioxane

Note: Other compounds may follow as required by clients due to regulatory changes and will be done in same manner as mentioned in SOP.

2.0 SUMMARY OF METHOD

- 2.1 Method 8260 is used to determine volatile organic compounds in samples by Purge-and-Trap (Method 5030/5035). The analytes are introduced directly onto a narrow-bore column that is directly interfaced to the ion source. The compounds are identified by retention times and comparing the mass spectra with the spectra of standards. The quantitative results are determined by comparing the response of a quantitative ion to an internal standard and using a minimum of a five-point calibration curve.
- 2.2 The low detection of 1,4-dioxane is now required by many laboratories due to State or Federal cleanup requirement to 2.7 ppb or lower. Due to the poor purge efficiency of 1,4-dioxane it is necessary to modify the traditional purge and trap (P&T) conditions and GC/MS Scan mode.
- 2.3 The P&T conditions have been modified by heating the soil or water sample to 60 C. This allows the 1,4-dioxane to efficiently transfer from the sample matrix to the trap followed by the release into the GCMS in the bake mode. The U-shaped geometry of the trap supplied with the Teledyne Tekmar Stratum Sample Concentrator aids in the efficient removal of water at the elevated sample purge temperature.
- 2.4 The GC/MS system required for low level 1,4-dioxane detection the system needs to be configured in the Selected Ion Monitoring (SIM) Mode. In the SIM Mode, the MS gathers data for masses of interest rather than looking for masses over a wide range. Typically the MS looks for masses of four or less ions per compound thus making the GC/MS-SIM scan 10 to 100 times more sensitive than the full Scan mode. The Agilent system ALF uses is able to perform SIM and SCAN

mode simultaneously resulting in detection of both a wide range of compounds and low level detection.

3.0 INTERFERENCE

3.1 Sample pathways can become contaminated if the pathway is exposed to samples with high levels of target analytes present. In the case where an analyte is present at a concentration of over 1000 ppb, samples following this should not be reported for this analyte until a blank or sample is analyzed and found to be below lowest quantitative value reported or “clean.”

4.0 DEFINITIONS

CCV- Continuing Calibration Verification- is a procedure to determine whether an instrument was within acceptable calibration throughout the period in which samples were analyzed (i.e., to verify that the initial calibration was applicable during the sample analyses).

COC- Chain of Custody- a record that documents the possession of the samples from the time of collection to receipt in the lab. This record generally includes the number and types of samples, the sampler, the collection time and date, and the requested analyses

CVS- Calibration Verification Standard- An intermediate concentration level standard prepared, analyzed and evaluated against the initial calibration curve. This standard is analyzed every 12 hours and must meet the acceptance criteria as noted in EPA Method 8260 Section 7.4.4 through 7.4.7

DOC- Demonstration of Capability- the procedure to establish the ability of an analyst to generate acceptable accuracy. May be Initial (IDOC) or Continuing (CDOC) procedure..

DOH- Department of Health

DI – De-ionized Water (may substitute any other contaminant free water)

GC/MS- Gas Chromatograph/Mass Spectrometer

IC- Initial Calibration as indicated in Section 9 of this SOP

ICB- Initial Calibration Blank- Blank analyzed following an initial calibration.

ICV- Initial Calibration Verification- similar to CVS but analyzed immediately following an initial calibration.

LCS - Laboratory Control Sample- A sample matrix free from the analytes of interest spiked with known concentrations of analytes of interest. Used to establish intra-laboratory precision and to assess the performance of the measurement system.

LOD- Limit of Detection. Equivalent to MDL.

LOQ- Limit of Quantitation. Equivalent to PQL.

MB- Method Blank. Equivalent to RB and Blank. A sample of a matrix similar to the batch of associated samples that is intended to contain none of the analytes of interest and which is processed with and under the same conditions as the associated samples

MDL – Method Detection Limit- Equivalent to LOD. MDL is the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero, and is determined from analysis of a sample in a given matrix containing the analyte.

MeOH – Purge and Trap Grade Methanol

MS- Matrix Spike- A selected sample from the analytical set spiked with the appropriate

test specific standard and surrogate.

MSD- Matrix Spike Duplicate- A second or duplicate selected sample from the analytical set spiked with the appropriate test specific standard and surrogate.

NA- Not Applicable

NELAC- National Environmental Laboratory Accreditation Conference- A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

NELAP- National Environmental Laboratory Accreditation Program- The overall program of which NELAC is a part.

ND- Not Detected - The value is less than the MDL.

PPB- parts per billion

PQL – Practical Quantitation Limit- Equivalent to LOQ. PQL is a quantitation limit that represents a practical and routinely achievable quantitation limit with a high degree of certainty (>99.9% confidence) in the results.

QA- Quality Assurance- An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets the defined standards of quality with a stated level of confidence.

QC- Quality Control- The overall system of technical activities whose purpose id to measure and control the quality of a product or service so that it meets the need of users.

RB- Reagent Blank. Equivalent to MB and Blank

RL – Reporting Limit

RPD- Relative Percent Difference- the difference between the amount measured and the true value, expressed as a percentage

SOP- Standard Operating Procedure- A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method of performing certain routine or repetitive tasks.

Standard Deviation- A computed measure of variability indicating the spread of the data set around the mean.

VOA- Volatile Organic Analysis or Volatile Organic Aromatics

VOC- Volatile Organic Compounds

VOH- Volatile Organic Halocarbons

5.0 METHOD DETECTION LIMITS

- 5.1 The method detection limit is defined as the minimum concentration of substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.
- 5.2 MDL's should be determined using the procedure as described in 40CFR Part 136 Appendix B. Prepare and analyze 7 replicate LCS at the concentration required to achieve necessary MDL's. Calculate the Standard Deviation and the Method Detection Limit using the Student "T" factor of 3.143 for 7 replicates.
- 5.3 The MDL's for this method are included in the tables 18.4 and 18.5 below.

6.0 EQUIPMENT, EQUIPMENT CONDITIONS AND SUPPLIES

6.1 Teledyne Purge and Trap Concentrator (PTC) – Stratum with 25 ml sparge tube. The sample heater assembly for all glassware types, 110V added accessory. PTC is plumbed with a #9 Type trap to aid in moisture control. An equivalent PTC system may be used.

6.1.1 PTC Conditions

1. Valve Oven Temperature:	150C
2. Transfer Line Temperature:	150C
3. Sample Mount Temperature:	90C
4. Purge Ready Temperature:	30C
5. Condenser Ready Temperature:	40C
6. Condenser Purge Temperature:	20C
7. Standby Flow:	2 mL/min.
8. Pre-Purge Time:	0.5 min.
9. Pre-Purge Flow:	5.0 mL/min.
10. Sample Heater:	On
11. Sample Preheat time:	4.0 min.
12. Sample Temperature:	60C
13. Purge Time:	5.0 min.
14. Purge Temperature:	0C
15. Purge Flow:	20 mL/min.
16. Dry Purge Time:	1.0 mL/min.
17. Dry Purge temperature:	25C
18. Dry Purge flow:	50 mL/min.
19. GC Start:	Start at Desorb
20. Desorb Preheat Temperature:	245C
21. Desorb Drain:	On
22. Desorb Time:	2.0 min
23. Desorb Temperature:	250C
24. Desorb flow:	300mL/min.
25. Bake Time:	2.00 min.
26. Bake Temperature:	270C
27. Bake Flow:	400mL/min.
28. Condenser Bake Temperature:	175C

6.2 GC/MS: HP5975C VL-MSD Triple Axis Detection with HP 7890A GC- J&W DB-VRX column 20meter, 0.18mm ID, 1um df). The hardware and software (Agilent Chemstation) allow for operation in the SIM Mode (Separately or Simultaneously with Scan Mode). An equivalent GC/MS-SIM system may be used.

6.2.1 GC Parameters

1. GC: Agilent 7890A or equivalent may be used.
2. Column: J&W DB-VRX column 20 M, 0.18MM ID, 1 um df or similar.
3. Oven Program: 45C for 6 min.; 18C/min. to 100C for 0 min; 25C to 200 for 0.9 min; 14 minute run time. (Note: Each GC/MS system may have a slightly different Oven program due to hardware and column differences).

4. Inlet: 200C; Inlet flow 24mL/min
5. Column Flow: 1.0 ml/min of Helium gas
6. Split Ratio: 30:1
7. Front Inlet Pressure: 21.03 psig
8. Auxiliary temperature: 225C

6.2.2 MS Detector Parameters

1. MSD: Agilent 5975C Inert XL or equivalent.
2. Source: 230C
3. Quad: 150C
4. Solvent delay: 0 min
5. Column flow: 1.0 mL/min
6. SIM Ions: Primary Ions 88 and 76
Secondary Ions 58 and 57
7. Dwell time: 100msec per ion

- 6.3 Syringes- Hamilton Gas Tight, 1ul-10m
- 6.4 Class A Volumetric Flasks, 50 ml
- 6.5 Balance- Acculab ProPocket-250B

7.0 REAGENTS AND STANDARDS

7.1 Organic Free Water

7.2 Methanol (MeOH), CH₃OH- Purge and Trap Grade

7.3 Stock Standards-Purchased as Certified Solutions

8.0 SAMPLE COLLECTION, SHIPMENT, HANDLING AND PRESERVATION

8.1 Water Samples-The sampling process should yield a minimum of 2 vials (20-40mls each), filled to the top, with no air bubbles present. The samples must be kept on ice until they have reached the lab and have been logged in. Once the samples have been logged in, they are kept in a refrigerator at 4 degrees Celsius. If the samples were not preserved during sampling the hold time for those samples is 7 days. If the samples were preserved (with Hydrochloric acid) the hold time is 14 days.

8.2 Soil Samples- The sampling process should yield 1 soil vial per sample. . The samples must be kept on ice until they have reached the lab and have been logged in. Once the samples have been logged in, they are kept in a refrigerator at 4 degrees Celsius. Samples hold time for those samples is 14 days.

9.0 CALIBRATION, STANDARDIZATION, PROCEDURES and CALCULATIONS

9.1 Tuning- Prior to analysis of samples or standards the Mass Spec must pass a BFB tune. To start the process, an tune is preformed through the **Instrument Control** screen (**GC/MS Instrument #1**). Choose the options below to tune the MS.

1. **View**
2. **Tune an Vacuum Control**
3. **Tune**
4. **BFB Tune**

The software will automatically start the MS tune. After completion of the tune, a report will be generated, and the results can be accepted according to the following criteria listed in the GC/MS software:

>> The mass assignments shown in the upper “profile” part of the display should be within +/- 0.2 amu of 69, 219, and 502.

>> The peak widths (PW) of these three peaks should be 0.5 +/- 0.1 amu.

>> The mass assignments shown in the lower “scan” part of the display should be within +/- 0.1 amu of 69, 219, and 502.

>> The relative abundance should show that the peak at 69 amu is the largest.

>> The Isotope mass assignments should each be 1 amu greater than the mass assignments of the parent peaks.

>> The Isotope ratio figures should be close to the theoretical values of 1.08 for m/z 69, 4.32 for m/z 219, and 10.09 for m/z 502.

>> If mass 28 is greater than mass 18, there may be an air leak in the system. Exceptions are when it is within 1hour of venting or during the first autotune after refilling the calibration vial.

Go to **File** and **Save Tune Values** to file **BFB1**. To return to the Instrument Controls choose **View** and then **Instrument Controls**.

Once the Autotune has passed, analyze a blank spiked with BFB at a concentration between 5-50ng. When BFB is chosen as the sample type in the sequence (at **MS Top**) the report will automatically print out and each parameter will be listed with a Pass or Fail. Once the BFB tune passes analysis can begin. In addition, a BFB tune should be performed every 12 hours while samples are being analyzed to ensure that all of the MS parameters met specified mass intensity criteria. As long as the BFB tune continues to pass every 12 hours, the tuning requirements have

been fulfilled. If the BFB tune fails, perform an Autotune and /or adjust MS parameters until a BFB tune passes. If the BFB tune continues to fail, it may be a sign that there is an instrument problem such as a dirty ion source. See Table 18.3 for Acceptance Criteria.

- 9.2 Quantitation Database-The Quantitation Database stores information about each compound including Retention Times, Target Ions, Curve Fit, Compound Attributes, and Calibration Levels. Go to the **Environmental Data Analysis** screen and under **Initial Cal** there is an option to **Set Up Quantitation**. Using a data file where all compounds are present, identify each compound and insert it into the Quantitation Database starting with the first internal standard. Second, enter the compound with the shortest retention time until all the compounds of interest are listed. After all of the compounds have been identified and entered into the database, additional information can be inserted as well. For example, Method 8260 recommends that certain compounds have specific functions or attributes (page 2 of the database). They are as follows:

Surrogate Standards- toluene-d8, 4-bromofluorobenzene,
1,2-dichloroethane-d4, and dibromofluoromethane

Internal Standards- fluorobenzene, chlorobenzene-d5, and
1,4-dichlorobenzene-d4

Tune Standard- 4-bromofluorobenzene

- 9.3 Water Calibration-Once the Quantitation Database has been prepared, a calibration curve can be analyzed and the levels updated. Make up to nine levels of standards that contain all of the compounds that are required for 8260 analysis. Note that all working standards are prepared in 50ml of deionized water and immediately transferred to a 40ml volatile vial for analysis. ALF also has prepared calibration standards by preparing working standards of 10-400 ng/ul and 100 – 4000 ng/ul in 2ml of methanol. The 1 – 50 ul volume of working methanol standards have then been placed in 10 ml of water preloaded into a syringe and loaded into the P&T sample concentrator.

- 9.3.1 Analyze the 5 to 9 working standards. Now there is sufficient information to plot the response of the compounds at different concentration levels. Go to the **Environmental Data Analysis** screen, open the file that corresponds to the first standard (ex: STD 4, 300 ppb) by selecting **File/ Load Data File**. Once the file is opened, select **InitCal/Update Levels**. Select **Existing Level ID**, **Update Level ID** and select 7 from the calibration level ID selection box. **Replace or Add Level** to the Responses and the Retention Times, and select **Do Update**. Repeat this step until each level is entered.

- 9.3.2 After all of the levels are entered, go to **InitCal/Edit Compounds** choose a compound and select **View**. On **Page 3**, all of the levels should be listed with the concentration, and response. Select **Plot** and view the Relative Standard Deviation for the Response Factor. An acceptable RF RSD is less than 20% with the exception of any compound specified to be a Calibration Check Compound (acceptable limits are less than 30%). View each compound, and make sure that the criterion for the RF has been met. Up to two points may be removed from the curve to bring the RF RSD into range. If the lowest level is removed from a compound, the detection limit will change, and if the highest point is removed the reporting limit will change. If an acceptable RF RSD can not be obtained for one or more compounds, with a 5-point calibration, Linear Regression or Quadratic may be used to determine if the calibration is acceptable for that compound. In addition, if the mean of the RSD value for all the compounds in the calibration is less than 20% the calibration is acceptable. The Response Factor is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Peak Area of the analyte or surrogate

A_{is} = Peak Area of the Internal Standard

C_s = Concentration of the analyte or surrogate

C_{is} = Concentration of the Internal Standard

- 9.3.3 Save the method after all necessary changes have been made. Go to **File/Save Method** and select **OK**.

9.4 Soil Calibration - Once the Quantitative Database has been prepared, a calibration curve can be analyzed and the levels updated. Make up to nine levels of standards that contain all of the compounds that are required for 8260 analysis. Working standards are prepared in 24 ml VOA vials containing approximately 20 - 22 ml of analyte-free water and 2 – 5 grams of soil (exact volumes and weights are recorded), capped and mixed three times. The calibration working standards are prepared with methanol working standards of 10ng/ul and 100ng/ul in 2ml of methanol. The 1 – 50 ul volume of working methanol standards have then been placed into 10 ml of soil extracted water preloaded into a syringe and loaded into the P&T sample concentrator.

- 9.4.1 Next, analyze up to nine working standards by following the instructions in the SOP **Method 5035-Purge-and-Trap Analysis for Soil Samples**. Now there is sufficient information to plot the response of the compounds at different concentration levels. Go to the **Environmental Data Analysis** screen, open the file that corresponds to the first standard (level

7, 100ppb) by selecting **File/ Load Data File**. Once the file is opened, select **InitCal/Update Levels**. Select **Recalibrate** and select **7** from the calibration level ID selection box. **Replace** the Responses and the Retention Times, and select **Do Update**. Repeat this step until each level is entered.

- 9.4.2 After all of the levels are entered, go to **InitCal/Edit Compounds** choose a compound and select **View**. On **Page 3** all of the levels should be listed with the concentration, and response. Select **Plot** and view the Relative Standard Deviation for the Response Factor. An acceptable RF RSD is less than 20% with the exception of any compound specified to be a Calibration Check Compound (acceptable limits are less than 30%). View each compound, and make sure that the criterion for the RF has been met. Up to two points may be removed from the curve to bring the RF RSD into range. If the lowest level is removed from a compound, the detection limit will change, and if the highest point is removed the reporting limit will change. If an acceptable RF RSD can not be obtained for one or more compounds, with a 5-point calibration, Linear or Quadratic Regression may be used to determine if the calibration is acceptable for that compound. In addition, if the mean of the RSD value for all the compounds in the calibration is less than 20% the calibration is acceptable. The Response Factor is calculated as follows:

$$\text{RF} = \frac{\text{As} \times \text{Cis}}{\text{Ais} \times \text{Cs}}$$

Where:

As = Peak Area of the analyte or surrogate

Ais = Peak Area of the Internal Standard

Cs = Concentration of the analyte or surrogate

Cis = Concentration of the Internal Standard

- 9.4.3 Save the method after all necessary changes have been made. Go to **File/Save Method** and select **OK**.
- 9.5 Sample Analysis- Samples are analyzed in batches. A batch consists of a Method Blank, a Laboratory Control Standard, a Matrix Spike, Matrix Spike Duplicate, and up to 20 samples. Batches must be specific to the sample matrix; waters and soils can not be in the same batch.
- 9.5.1 GC/MS calibration verification- Consists of 3 steps that are performed at the beginning of each 12-hour analytical shift.
- 9.5.1.1 Prior to the analysis of samples or calibration standards, introduce 5-50ng of 4-bromofluorobenzene (BFB) standard into the GCMS. The resultant mass spectra for the BFB must meet the criteria in Table 18.3 before sample analysis begins. This criteria must be

demonstrated each 12-hour shift during which samples are analyzed.

9.5.1.2 The initial calibration curve for each compound of interest should be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing a CCV. The results should meet the verification acceptance criteria provided. Note: The BFB and CCV may be combined into a single standard as long as both acceptances criteria can be met with out interference.

9.5.1.3 A method blank should be analyzed during the calibration verification procedure or at any other time during the analytical shift to ensure that the total system is free of contaminants. If the method blank indicates contamination, reanalyze in the event the contamination is carry over from a previous sample. See Section 8.0 of Method 8000 for method blank performance criteria.

9.5.2 Set up samples in the desired order of analysis in the Teledyne Stratum Purge and Trap Concentrator. Set up the method to be analyzed on the concentrator using the front control panel. Then create a sequence as listed below so that the GC software will collect data from the Mass Spec.

9.5.3 To run a sequence go to the **GC/MS Instrument #1 MS Top/Environmental** screen.

1. Select **Method**, then **Load**.
2. The current method being used is **8260.M**
3. **OK**
4. Select **Sequence/Edit Sample Log Table**
5. Fill in the Log Table.
6. Select **OK**
7. Select **Save**
8. Save the file as **DDMMYY.S**
9. Select **OK, Run**, and **Run Sequence**.

9.6 Acquiring Data-As the GC/MS is running a sample, it can be viewed through the Environmental Data Analysis screen. Go to File/Take Snapshot. This option can only be used when a sample run is in progress; once the sample run is completed the file can only be viewed by File/ Load Data File. Quantitation can take place with both of these options. Select Quant/ Calculate/ Generate Report. Then the option will be given to show the results on the screen and/or print the results. Once the options have been chosen, select OK. At this point, the data should be collected and reviewed for reporting.

9.7 Calculations

Dry weight determination:

$$\text{mg/dry kg PH} = \frac{C_s}{1 - (\% \text{moisture}/100)}$$

Where: C_s = Concentration of Petroleum Hydrocarbons (mg/L or mg/kg)

Matrix Spike Recovery

$$\% \text{ Recovery} = [(A - B) / C] \times 100$$

Where: A = measured concentration of spiked sample

B = measured concentration of sample

C = actual spike concentration

Duplicate Precision

$$\text{RPD} = \frac{A}{B} \times 100$$

Where: A = Absolute value of Dup 1 – Dup 2

B = average of Dup 1 and Dup 2

LCS Recovery

$$\% \text{ Recovery} = (A / B) \times 100$$

Where: A = measured concentration of spiked sample

B = actual concentration of sample

Surrogate Recovery

$$\% \text{ Recovery} = (A / B) \times 100$$

Where: A = concentration found

B = concentration added

10.0 DATA ASSESSMENT and ACCEPTANCE CRITERIA

- 10.1 Reviewing Data-All the data should be reviewed thoroughly to ensure that it is as accurate as possible. To begin the Method Blank must be “clean” or below the reporting limit. The LCS should have a percent recovery +/- 30%. The samples Internal Standard recovery must be between 50% and 200% of the Internal Standard recovery in the Laboratory Control Standard. Once these Quality Control parameters have been met, the data can be reviewed in closer detail to determine if the results are reportable or if the sample should be reanalyzed. Begin by using the CVS Quantitation Report as a reference to compare sample Quantitation Reports. As stated above, make sure that the Internal Standard recoveries are within QC limits (50%-200%). In addition, it is important to verify that the Retention Times of each analyte are within 0.1 minutes of the CVS

Retention Time for that analyte. The Qvalue should also be used to determine if the result of a particular compound is reportable. Currently, the analyte results are considered reportable if the Qvalue is over 80, but exceptions are made if there appears to be matrix interference and/or the chromatogram is examined manually. Below is the definition of the Qvalue as described by the **HELP** section of the software:

The Qvalue is an approximate measure of the similarity between the expected intensities of the quantitation and qualifier ions and the actual intensities. The Qvalue should not be construed as an absolute measure of the quality of the closeness of fit. The Qvalue only examines the intensities of the quantitation ion and the qualifying ions. When a compound has no qualifier ions, the Qvalue will always be 100.

- 10.2 Next, examine the concentration of each analyte. The concentration (in ppb) is not reportable below the Method Detection Limit and over 20% above the highest point on the calibration curve. If any analyte in a sample is outside any of these parameters, the sample should be reanalyzed (possibly at a different dilution) to achieve reportable results. Samples were analytes greater than 20% above the range of calibration may be reported if the proper qualifiers are used.
- 10.3 All results should be recorded on the GC/MS Volatiles Bench sheet or on the original chromatograph. These results may be used in reporting or as a reference depending on whether or not the sample met the appropriate QC requirements (listed above).
- 10.4 Reporting Data- Once all of the results for a specific work order are complete, the results can be reported on an Excel spreadsheet. Templates for the Excel spreadsheets are saved in Windows Explorer on the C drive. To locate a template, go to Start, Programs, Windows Explorer, and select the method to be reported (ex: 8021b Water). Use the information provided in the Log In sheet and the GC Bench sheet to fill in the Excel template. As the results are being transferred from the GC Bench sheet to the Excel spreadsheet, make sure that any analyte results above the MDL (currently 1 ppb) are highlighted using the Bold option and reported using two significant digits. When the template has been filled out completely, go to File, Save As, and use the Work Order number to save the template as a results file.

Please reference NELAC Quality Systems Chapter 5 Appendix C and D for additional guidance of acceptance criteria.

11.0 QUALITY CONTROL

- 11.1 Refer to Chapter One and Method 8000 for specific quality control procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.
- 11.2 Quality control (QC) procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:
- 11.2.1 The GC/MS system must be tuned to meet the BFB specifications. In Secs. 7.3.1 and 7.4.1 (See Tables, diagrams,... of this SOP for passing criteria)
 - 11.2.2 There must be an initial calibration of the GC/MS system as described in Sec 7.3.
 - 11.2.3 The GC/MS system must meet the SPCC criteria in Sec 7.4.4 and CCC criteria in Sec 7.4.5 each 12 hours of analysis.
- 11.3 Initial Demonstration of Capability (DoC) – Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec 8.0 for information on how to accomplish this demonstration.
- 11.3.1 Acceptance Criteria – For the Initial and Continuing Demonstration of Capability to be acceptable, the mean recovery for all compounds should be between 70 – 130% and the Standard Deviation less than 20%.
 - 11.3.2 Table 18.6 summarizes the initial DOC for the EPA8260 SIM Method.
- 11.4 Sample Quality Control for Preparation and Analysis – The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.
- 11.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank

- 11.4.2 should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.
 - 11.4.3 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.
 - 11.4.4 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
 - 11.4.5 See Method 8000, Sec 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.
- 11.5 Surrogate recoveries – The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.
- 11.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the Calibration Verification Standard (CVS) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is performing acceptably, the injector is leaking, the injection septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.
- 11.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

- 11.8 Biannual LCS Check- As indicated in NELAC Quality Systems Chapter 5 appendix D. For test methods that have extremely long lists of analytes, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period. Acceptance criteria same as LCS
- 11.9 Limit of Detection- The laboratory shall determine the LOD for the method for each target analyte of concern in the quality system matrices. Acceptance criteria same as LCS
- 11.10 Limit of Quantitation- The laboratory shall determine the LOQ for each analyte of concern according to a defined documented procedure. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. Acceptance criteria same as LCS.

For additional assistance, please reference NELAC Quality System Chapter 5 Appendix C.

- 11.11 Proficiency Testing- Proficiency testing is performed in accordance to NELAC requirements. The results are also used as intra-laboratory examples if continuing demonstration of performance.

12.0 METHOD PERFORMANCE

- 12.1 This method has been tested using purge –and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore column, water was spiked at concentrations between 0.5 and 10 ug/l. Single laboratory accuracy and precision data are presented for the method analytes in the tables.
- 12.2 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in soil (sand) matrices. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 20ug/kg. These data is listed in the tables.

13.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Corrective actions for out-of-control or unacceptable data may include consultation with the QA Officer and/or the Lab Director. No list can anticipate every possible corrective action. The following is a list of the most commonly performed corrective actions. Ultimately, consultation with the client is recommended if these corrective actions fail to produce satisfactory results.

- 13.1.1 Sample reanalysis
- 13.1.2 Sample re-extraction
- 13.1.3 Instrument Maintenance

- 13.1.4 Evaluation of calibration curve, including continuing calibration verification and/or initial calibration.
- 13.1.5 Notification of client

14.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL DATA

- 14.1 As noted in 13.0, the ultimate contingency for handling out-of-control or unacceptable data is consultation with the client. There are situations with some sample matrices which will not produce data with the confidence that is achievable with other samples. When corrective actions fail to correct a problem with unacceptable or out-of-control data, the results shall be discussed with the client by the QA Officer and/or the Lab Director.

15.0 SAFETY

- 15.1 The toxicity or 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. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. All personnel should wear gloves, a lab coat, safety goggles, long pants, and completely encasing shoes to help ensure safety.

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

- 16.1 All laboratories generate wastes. Some wastes can be hazardous such as acidic wastes, metal bearing wastes and organic wastes.
 - 16.1.2 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.
 - 16.1.3 Non-Hazardous waste with other pH levels may be directly poured in the sink.

17.0 REFERENCES

1. Method 8260-Volatile Organic Compounds By Gas Chromatography/ Mass Spectrometry (GC/MS), U.S. Environmental Protection Agency, Revision 2, December 1996.
2. Method 8000-Determinative Chromatographic Separations, U.S. Environmental Protection Agency, Revision 3, December 1996.

3. Method 5030B-Purge and Trap for Aqueous Samples, U.S. Environmental Protection Agency, Revision 2, December 1996.
4. Method 5035-Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, U.S. Environmental Protection Agency, Revision 0, December 1996.
5. Analysis of Selected Halogenated and Aromatic Volatile Organic Compounds by GC in Water and Soil Matrices. Document 04001R0, Analytical Laboratories of Florida April 2004.
6. NELAC Quality Manual for Analytical Laboratories of Florida, Revision 2, November 2006.
7. Analysis of 1,4-Dioxane Using the Stratum PTC and SOLATek 72, Teledyne Tekmar 1,4-Dioxane Application Note, July 9, 2010.

18.0 TABLES, DIAGRAMS FLOWCHARTS, VALIDATION DATA

18.1 Water Calibration Curve Preparation and Concentration made in 50ml volumetric flask

Level	Conc.	Cal Std	Water Vol.	IS added	Amount Present	GC/MS Description
Level 9	1000 ppb	1000 ul	50 ml	5.0 ul	1000 ppb	STD 9
Level 8	500 ppb	500 ul	50 ml	5.0 ul	500 ppb	STD 8
Level 7	300 ppb	300 ul	50 ml	5.0 ul	300 ppb	STD 7
Level 6	100 ppb	100 ul	50 ml	5.0 ul	100 ppb	STD 6
Level 5	30 ppb	30 ul	50 ml	5.0 ul	30 ppb	STD 5
Level 4	20 ppb	20 ul	50 ml	5.0 ul	20 ppb	STD 4
Level 3	10 ppb	10 ul	50 ml	5.0 ul	10 ppb	STD 3
Level 2	3 ppb	3 ul	50 ml	5.0 ul	3 ppb	STD 2
Level 1	1 ppb	1 ul	50 ml	5.0 ul	1 ppb	STD 1
Level CCV	10ppb	10 ul	50 ml	5.0 ul	100 ppb	CCV1

Note: Working Methanol cal std concentration is 50 ng/ul of 1,4-dioxane for 50ml volumes and 10ng/ul for 10 ml volumes.

18.2 Soil Calibration Curve Preparation and Concentration made in 24 ml soil vials

Soil Curve		Cal Std	Soil Vol.	IS added
STD 9	10000ng or 4760 ppb	20 ml H2O + 4.2g soil 1000 ul	10 ml	5ul
STD 8	5000ng or 2380ppb	20 ml H2O + 4.2g soil 500 ul	10 ml	5ul
STD 7	3000ng or 1430 ppb	20 ml H2O + 4.2g soil 300 ul	10 ml	5ul
STD 6	1000ng or 476 ppb	20 ml H2O + 4.2g soil 100 ul	10 ml	5ul
STD 5	300ng or 143 ppb	20 ml H2O + 4.2g soil 30 ul	10 ml	5ul
STD 4	200ng or 95.2 ppb	20 ml H2O + 4.2g soil 20 ul	10 ml	5ul
STD 3	100ng or 47.6 ppb	20 ml H2O + 4.2g soil 10 ul	10 ml	5ul
STD 2	30ng or 14.3 ppb	20 ml H2O + 4.2g soil 3 ul	10 ml	5ul
STD 1	10ng or 4.8 ppb	20 ml H2O + 4.2g soil 1 ul	10 ml	5ul
Level CCV	100ng or 47.6 ppb	20 ml H2O + 4.2g soil 10 ul	10 ml	5ul

Note: Each calibration curve will vary slightly the soil sample size or water extract volume. Calibration Standard concentration is 10 ng/ul 1,4-dioxane in methanol.

$$\text{Concentration (ppb)} = \frac{(\text{loaded ng amount})}{(4.2 \text{ grams sample size})} \times \frac{(20 \text{ ml total extract volume})}{(10 \text{ ml sample extract})}$$

18.3 BFB Tuning Acceptance Criteria

Target Mass	Relative to Mass	Lower - Upper Acceptance %
50	95	8 - 40
75	95	30 - 60
95	95	100 - 100
96	95	5 - 9
173	174	0.00 - 2
174	95	50 - 200
175	174	5 - 9
176	174	93 - 101
177	176	5 - 9

18.4 MDL Study for EPA 8260 SIM Compounds in Water

WATER

<u>Compound</u>	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Run 4</u>	<u>Run 5</u>	<u>Run 6</u>	<u>Run 7</u>	<u>S. D.</u>	<u>mdl(ppb)</u>
1. 1,4-Dioxane	5.28	3.27	3.93	3.76	5.03	3.54	3.81	0.703	2.21

Note(s): MDL was determined with 7 replicates of 3.0 ppb in water.

Mdl = 3.143 x S.D.

Initial MDL Study performed on 2/19/11

18.5 MDL Study for EPA 8260 SIM Compounds in Soil

SOIL

<u>Compound</u>	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Run 4</u>	<u>Run 5</u>	<u>Run 6</u>	<u>Run 7</u>	<u>S. D.</u>	<u>mdl(ppb)</u>
1. 1,4-Dioxane	16.73	18.73	20.86	17.75	16.2	17.49	18.01	1.52	4.8

Note(s): MDL was determined with 7 replicates of 14.3 ppb in soil.

Mdl = 3.143 x S.D.

Initial MDL study performed on 2/19/11

18.6 Initial DoC Study for EPA 8260 SIM Compounds in Water

Water

Compound	Run 1	Run 2	Run 3	Run 4	Average	% Rec.	S. D.
1. 1,4-Dioxane	23.02	25.53	25.46	27.89	25.47	127.4	1.99

Note(s): DoC was determined with 4 replicates of 20 ppb in 10 ml water volume.
Initial DoC date 2/19/11



State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E83934

ANALYTICAL LABORATORIES OF FLORIDA
166 CENTER STREET, SUITE 111 1FDKE37F9SHA38915 (MOBILE
LAB)
CAPE CANAVERAL, FL 32920

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2011 Expiration Date: June 30, 2012



A handwritten signature in black ink.

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E83934-13-07/01/2011
Supersedes all previously issued certificates

APPENDIX D

**EMPIRICAL LABORATORIES, INC.
STANDARD OPERATING PROCEDURES AND CERTIFICATION**

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 21 EFFECTIVE DATE: 20100901

METALS DIGESTION/PREPARATION

References:

Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C 21st
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director:  Date: 9/8/10

Data Quality Manager:  Date: 9/8/10

Section Supervisor:  Date: 9/9/10

Changes Summary

Revision 21, 9/1/10

- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:

**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
 - a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
 - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
 - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
 - a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. NOTES:

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

- A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO₃ to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO₃ is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS

1. Samples are taken in high density polyethylene (CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS

1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
- B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
- C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

- A. Fume hood, Labconco or equivalent.
- B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.
- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
- D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
- E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.

- F. Beckman CS-6R centrifuge.
 - G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
 - H. Whatman No. 41 filter paper or equivalent.
 - I. Whatman No. 42 filter paper or equivalent.
 - J. Whatman 0.45 micron filter paper or equivalent.
 - K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
 - L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
 - M. Manual Sample Mill
 - N. Wiley Sample Mill
 - O. Clippers for cutting vegetation
- NOTE:** All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO₃). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H₂SO₄). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

- a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in LIMS.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIIM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample LIMS.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO₃ and 5% HCl. Use 0.125 mLs HNO₃ and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

- a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. BS1 and then BS2 etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbook and transferred to LIMS.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

IX. PROCEDURE

- A. Glassware preparation for digestion or when the hot-block can not be used:
1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
 2. Rinse glassware with reagent water that contains 5% HNO₃ and 5% HCl followed by a rinse with reagent water.
 3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.
- B. Aqueous sample filtration (for dissolved metals):
1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO₃ followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
 2. Rinse a 0.45 micron filter with 1:5 HNO₃ thoroughly, followed by D.I. water.
 3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
 4. Discard the first 50 to 100 mL.
 5. A preparation blank must be taken through the filtration step and analyzed with the sample.
 6. Preserve the sample with HNO₃ to pH<2.
 7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO₃. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**
- C. Aqueous sample preparation
1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".
 - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
 - b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample.
 - c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, (12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
 - f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

- g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
 - h. The sample is now ready for analysis.
 - i. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
2. Method 200.7, "**Acid digestion procedure for total recoverable metals**".
 - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
 - b. Add 1.0 mL concentrated HNO₃ to the sample.
 - c. Add 2.50 mL concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
 - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
 - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
 - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - i. The sample is now ready for analysis.
 - j. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
 - a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
 - b. Add 1.5 mL concentrated HNO₃ to the sample.
 - c. Cover the sample with a ribbed watch glass.
 - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.
 - e. Add another 1.5 mL portion of concentrated HNO₃ to the sample.
 - f. Cover the sample with a ribbed watch glass.
 - g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

- h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
 - i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
 - j. Cover the digestion vessel and reflux for an additional 15 minutes.
 - k. Bring sample to its predigestion volume in digestion vessel.
 - l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
 - m. The sample is now ready for analysis.
 - n. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
 - b. Add 2.5 mL 1:1 HCl to the sample.
 - c. Heat 15 minutes in a hot bath.
 - d. Filter through a membrane filter.
 - e. Transfer to ICP analyst.
- D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry

enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"

- a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
- b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3 , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
- c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
- d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
- e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3 , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3 . The sample is now ready for analysis.
- f. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, "**Acid digestion of Sediments, Sludges and Soils**"

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

- c. Add 5 mL D.I. water and 5 mL concentrated HNO_3 (1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for

10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook

- d. Allow the sample to cool. Add 5 mL concentrated HNO₃, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO₃, repeat this step (addition of 5 mL of concentrated HNO₃) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO₃. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C ± 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H₂O₂ if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H₂O₂ to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H₂O₂.)
- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
- g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
- h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.
NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.
- j. The sample is now ready for analysis.
- k. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank
 - a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
 - b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
2. Blanks
 - a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
 - b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
 - c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
 - d. Sample is given a unique identifier in the digestion log.
3. Laboratory Control Samples
 - a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
 - b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
 - c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
 - d. Sample is given a unique identifier in the digestion log.
4. Duplicates
 - a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.
5. Blank Spike
 - a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike
 - a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.
 - b. The following metals do not get digested spikes when using CLP spike.
 - Calcium
 - Magnesium
 - Sodium

Potassium

- c. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)
- d. **The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
 1. Redigest another sample aliquot.
 2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
 1. All metals glassware must be kept separate from all other laboratory glassware.
 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.
- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
- F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

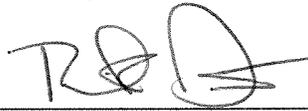
**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 103 REVISION #: 18 EFFECTIVE DATE: 041110

**MERCURY ANALYSIS IN WATER
BY MANUAL COLD VAPOR TECHNIQUE
METHODS USEPA SW846 7470A and 245.1 CLP-M 4.1
(NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003),
ADDENDUM FOR USEPA CLP ILM 05.2**

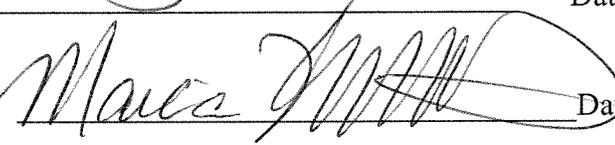
APPROVALS:

Lab Director:



Date: 4/18/10

Data Quality Manager:



Date: 4/11/10

Section Supervisor:



Date: 4/13/10

Changes Summary

Revision 18, 04/11/10

- The SOP is an update from Revision 17 dated 03/25/10
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Tables have been updated to reflect the current limits/processes.

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17. Pollution Prevention
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1.0 Identification of the Test Method

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury, and is compliant with SW846 Method 7470A, USEPA Method 245.1, and USEPA SOW ILM04.1.

2.0 Applicable Matrix or Matrices

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

3.0 Detection Limit

Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	AQUEOUS MDL/DL (ug/L)	AQUEOUS LOD (ug/L)	AQUEOUS ERL/LOQ (ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)
Mercury	0.080	0.16	0.20	0.20	0.20

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	253.7

4.0 Scope of Application, Including Components to Be Analyzed

- 4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.
- 4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

- 4.3 In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.
- 4.4 The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.
- 4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

6.0 Definitions

- 6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.
- 6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

- 7.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.
- 7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.
- 7.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).

- 7.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized and organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

8.0 Safety

- 8.1. Normal accepted laboratory practices should be followed while performing this procedure.
- 8.2. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- 8.3 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.
- 8.4 The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

9.0 Equipment & Supplies

- 9.1 Perkin Elmer Flow injection Mercury system.
- 9.2 Mod Block Digester set to maintain $95 \pm 2^\circ\text{C}$ for 2 hours.
- 9.3 Polypropylene sample digestion vessels with snap or screw caps or equivalent.
Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.

10.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra Scientific and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at room temperature.

10.1 REAGENTS

- 10.1.1 Concentrated sulfuric acid suitable for Hg determination.
- 10.1.2 Concentrated nitric acid suitable for Hg determination.
- 10.1.3 Stannous chloride: In a 1000 mL volumetric flask add approximately 500 mLs D.I. water, 30 mLs concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
- 10.1.4 3% HCl Carrier Solution: Dilute 30 mLs of concentrated metals grade HCl to one liter. Prepare fresh daily.
- 10.1.5 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.
- 10.1.6 Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mLs of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring (minimum of 3 hours if absolutely necessary). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.
- 10.1.7 Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mLs D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

10.2 STANDARDS

10.2.1 Traceability

- 10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within Element as well as on the container's label.
- 10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- 10.2.1.3. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All standards, blanks, and samples are taken through the digestion process.
- 10.2.1.4 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique Element identifier.

10.2.1.5 Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO₃. This solution is recorded in Element and given a unique Element identifier.

10.2.2 Calibration Standards

Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

10.2.2.1 Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.)

<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 30 mLs</u>
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

10.2.2.2 Appropriate reagents are added as below in the sample preparation section.

10.2.2.3 Prepare one vessel for each.

10.2.2.4 It is necessary to digest the calibration standards.

10.2.3 Calibration Verification Standards

10.2.3.1. Initial calibration verification (ICV) solution – 4.0 ug/L

10.2.3.1.1 Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

10.2.3.1.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.1.3 It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.3.2 Continuing calibration verification (CCV) solution

10.2.3.2.1 Prepared from the primary source standard.

10.2.3.2.2 Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

10.2.3.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.3.2.4 It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.4 Digestion standards

10.2.4.1 Blank Spike

10.2.4.1.1 Prepared from the secondary source standard.

10.2.4.1.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 This solution should be given a unique identifier within Element.

10.2.1.2 Matrix Spikes

10.2.1.2.1 Prepared from the secondary source working standard.

10.2.1.2.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

10.2.1.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.

11.2 The holding time for the mercury digestion is 28 days from time of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BS’s are analyzed at 0.10ug/L. See [Table 2](#) for acceptance criteria.

12.3 **(Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)**

12.4 Daily

- 12.4.1. **The instrument must be calibrated daily for all projects.**
- 12.4.2 Begin each analysis with an ICV(QCS) second source. The control limits are $\pm 10\%$ and IPC (CCV) for 245.1, limits are $\pm 5\%$ and subsequent analyses are $\pm 10\%$.
- 12.4.3 Analyze ICB. Control limits ($<\pm MDL$ for USACE or $\pm RL/CRDL$ for others and CLP), depending on method. **No analyte detected $>2xMDL$ for DOD.**
- 12.4.4 If the ICV (QCS) is not in control a new curve must be analyzed prior to sample analysis.
- 12.4.5 If the IPC (initial CCV) for 245.1 is not within the limits of $\pm 5\%$, try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.
- 12.4.6 Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are $\pm 20\%$ for SW846-7470 and $\pm 10\%$ for 245.1.
- 12.6.7 A CCB must always follow a CCV, the control limit is ($<\pm MDL$ for USACE or $\pm RL/CRDL$ for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected $>2xMDL$ for DOD.**
- 12.5 Quarterly or as needed when doing straight CLP work.
 - 12.5.1 IDL's for CLP 4.1.
- 12.6 Digestion
 - 12.6.1 BS data should be maintained and available for easy reference or inspection.
 - 12.6.2 BLK ($<1/2 \pm RL$ or $\pm RL/CRDL$ for common contaminants (DOD) and $\pm RL/CRDL$ for others and CLP).
 - 12.6.2.1 Employ a minimum of one preparation blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be re-digested and re-analyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.
 - 12.6.3 Laboratory control sample (BS)
 - 12.6.3.1. Employ a minimum of one laboratory control sample (BS) per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are $\pm 20\%$ for SW846-7470 and $\pm 15\%$ for 245.1. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.
- 12.7 Sample matrix:

- 12.7.1 Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. CLP does not allow this. Project specific requirements will take precedence in these situations.
- 12.7.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within $\pm 25\%$ of the true value (**$\pm 20\%$ for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.
NOTE: For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.
- 12.7.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of $\pm 20\%$ RPD shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.
- 12.7.4 For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be $\pm 10\%$. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.
- 12.7.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

13.0 Calibration and Standardization

Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1 Set up the instrument with proper operating parameters.

13.1.1 Perkin Elmer Flow Injection Mercury System (FIMS).

13.1.1.1. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. (**Perkin-Elmer tygon tubing, waste**

and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

- 13.1.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
- 13.1.1.3 Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
- 13.1.1.4 Turn on computer and go to icon "AA Win LAB Analyst".
- 13.1.1.5 Go to method; select "Hg CAL 2" then OK.
- 13.1.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.
- 13.1.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.
- 13.1.1.8 Save entered sample list under "Savesample info file" Note: description and batch ID are normally the date of analysis.
- 13.1.1.9 Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.
- 13.1.1.10 Turn the printer on.
- 13.1.1.11 Connect all tubing to the pump and blocks.
- 13.1.1.12 Start the pump by going to "FIAS" and click the pump 1 Icon (120).
- 13.1.1.13 The pump will start, then lock down and tighten the tubes onto the pump.
- 13.1.1.14 Turn on the nitrogen tank, it should be above 500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.
- 13.1.1.15 The pressure gauge on the PE100 should be just below 100.
- 13.1.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.
- 13.1.1.17 Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.
- 13.1.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl₂. (Click the valve fill inject and make sure flow is correct and the line is rinsed).
- 13.1.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.
- 13.1.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.
- 13.1.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".
- 13.1.1.22 "Select Location" enter #'s to be ran, and then press "OK". Samples are done in increments of 10 samples

13.2 Analyze the calibration standards as below.

- 13.2.1 New calibration points must be analyzed when the ICV analysis is not within $\pm 5\%$. **A curve must be analyzed daily for all projects especially USACE and CLP projects.**

- 13.2.2 The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of ≥ 0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

14.0 Procedure

14.1 Glassware preparation

14.1.1 After use, samples are neutralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.

14.1.2 Acid clean the glassware used for mercury prep as follows:

14.1.2.1 Rinse with low Hg content 1:1 HCl.

14.1.2.2 Rinse with D.I. water.

14.2 Label the vessels indicating which sample will be in each.

14.3 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below.

14.4 Sample preparation

14.4.1. Transfer 30 mLs, or an aliquot diluted to 30 mLs of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

NOTE: Normally, an automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix gets spiked. Check with your manager.

14.4.2 Add 1.5 mLs of concentrated sulfuric acid to each vessel and mix.

14.4.3 Add 0.75 mL of concentrated nitric acid to each bottle and mix.

14.4.4 Add 4.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mLs). If the purple color does not persist after the addition of 7.5 mLs KMnO_4 the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

NOTE: The same amount of KMnO_4 added to the samples should be present in the standards and blanks.

14.4.5 Add 2.4 mLs of potassium persulfate to each vessel and mix. Cover.

14.4.6 Heat for 2 hours in the block digester at $95 \pm 2^\circ\text{C}$ (the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool.

14.4.7 Samples may be saved at this point if there is not time to run the whole set that day.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

14.5 Sample analysis

14.5.1 Set up the instrument as described in the calibration section above.

14.5.2 When ready to run samples, add 1.8 mLs of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of $\pm 10\%$ for SW846-7470 and $\pm 5\%$ for 245.1. Followed by the ICB ($< \pm \text{MDL}$ for USACE or $\pm \text{RL/CRDL}$ for others and CLP).

14.5.3 Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of $\pm 20\%$ for SW846-7470 and $\pm 10\%$ for 245.1.

14.5.4 CCB must always follow the CCV. Control limits are ($< \pm \text{MDL}$ for USACE or $\pm \text{RL/CRDL}$ for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected $> 2 \times \text{MDL}$ for DOD.**

14.5.5 The auto-sampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ-ICV
10	SEQ-ICB
11	BS

AS LOC	Sample ID
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB

23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.6 Data Reporting

14.6.1 Reduce data to result which will be reported.

14.6.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Apply a least squares fit to the calibration standards plotting $\mu\text{g Hg/L}$ versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume (the 0.1 $\mu\text{g Hg}$ standard will be input as 1.0 $\mu\text{g Hg/L}$) (0.1 $\mu\text{g Hg}$ / 0.030 L solution).

15.3 Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.

15.4 Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.

15.5 Report the data as $\mu\text{g Hg/L}$ of sample.

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.3 mL of the second source standard to 30 mLs with reagent water in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

19.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

19.2.1 ICV (QCS for 245.1)- second source not within $\pm 10\%$.

- A. If the problem is with the solution, re-prepare, obtain new stock if necessary.
- B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within $\pm 20\%$ for SW846 and $\pm 10\%$ for (245.1, $\pm 5\%$ for initial IPC and $+ 10\%$ for subsequent IPCs)

- A. If the problem is with the solution, re-prepare, obtain new stock if necessary.
- B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.
 - 1. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
 - 2. If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 CORRECTIVE ACTION: DIGESTION RELATED

19.3.1 The preparation blank less than $<1/2$ RL or \pm RL/CRDL for common contaminants (DOD) and \pm RL/CRDL for others and CLP.

- A. If the problem is with the instrument or stannous chloride.
Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, re-prepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.
- B. If the problem is with the digestion.
All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be re-digested/re-analyzed or reported as qualified. The project manager or QA manager will make this determination.
- C. LCS not within control limits (or $\pm 20\%$, $\pm 15\%$ for **245.1**).
 - 1. If the problem is with the instrument, reanalyze when instrument is in control if further sample bottles are available.
 - 2. Is the problem is with the digestion.
 - a. If biased low, associated samples must be re-digested.
 - b. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

19.4.1 Replicate analysis RPD not within $\pm 20\%$

The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within $\pm 25\%$ (**$\pm 20\%$ for DOD projects**)

- A. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
- B. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

19.4.3 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage

during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

- 21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 7470A.*
- 21.2 *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B.*
- 21.3 *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.
- 22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.
- 22.3 Table 3, Technical Completeness / Accuracy Checklist
- 22.4 Table 4, Data Reviewers Checklist
- 22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

ADDENDUM FOR USEPA SOW ILM05.2

1. The CCV concentration must be different from the ICV.
2. The same CCV shall be used throughout analysis for an SDG.
3. Calibration standards must be within 5% of the standard concentration.
4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is $\pm 30\%$.
5. Spike samples at 1 ug/L for water.

Table 1

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	AQUEOUS MDL/DL (ug/L)	AQUEOUS LOD (ug/L)	AQUEOUS ERL/LOQ (ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)
Mercury	0.080	0.16	0.20	0.20	0.20

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	<ul style="list-style-type: none"> Daily ICAL prior to sample analysis Low standard at the RL/LOD level 	<ul style="list-style-type: none"> If more than one calibration standard is used, $r \geq 0.995$ Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-run curve Check instrument for maintenance needs <p>Samples cannot be analyzed until there is a passing calibration</p>
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Must be within $\pm 10\%$ of true value	<ul style="list-style-type: none"> Re-run ICV Repeat ICAL
Continuing calibration verification (CCV)	<ul style="list-style-type: none"> After every 10 field samples and at the end of analysis sequence. 	<ul style="list-style-type: none"> $\pm 20\%$ of true value 	<ul style="list-style-type: none"> Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.
Method Blank (BLK)	One per prep 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 $> RL$	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Notify the PM for further action Re-prep of samples associated with the BLK NCR will be required for data reported
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-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.
BS	One per prep batch	Most stringent criteria listed within the LIMS.	<ul style="list-style-type: none"> Re-analyze to confirm failed. Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. NCR will be required for data reported
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Average percent recovery should be between 80-120%, with a 20% standard deviation. 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis
MDL Study	Once per year	<ul style="list-style-type: none"> • Calculated value must be less than the Spike level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	<ul style="list-style-type: none"> • Parameter must be detected • the response must be 3-times the noise level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	<ul style="list-style-type: none"> • Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	Sequence ID:
Method: 7470A/245.1 (Mercury)	

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did BS meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation Blank (BLK) below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____

- 10. Sample preparation information is correct and complete. _____
- 11. Analytical results are correct and complete. _____
- 12. The appropriate SOP's have been used and followed. _____
- 14. "Raw data" including all manual integration's have been correctly interpreted. _____
- 15. "Special" sample preparation and analytical requirements have been met. _____
- 16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete. _____

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 104 REVISION #: 19 EFFECTIVE DATE: 041110

**MERCURY ANALYSIS IN SOIL/SEDIMENT
BY MANUAL COLD VAPOR TECHNIQUE
METHODS SW846 7471A 7471B, EPA 245.5 AND CLPILM 04.1
(NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003),
ADDENDUM FOR USEPA CLP ILM 05.2**

APPROVALS:

Lab Director:  Date: 4/12/10

Data Quality Manager:  Date: 4/11/10

Section Supervisor:  Date: 4/13/10

Changes Summary

Revision 19, 04/11/10

- The SOP is an update from Revision 18 dated 03/25/10.

Revision 18, 03/08/10

- The SOP is an update from Revision 17 dated 01/29/09.
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.
- Numerous improvements/modifications were made to this SOP. Details/specifications were added that require evaluation from start to finish.

Table of Contents

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2. Applicable Matrix or Matrices
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1.0 Identification of the Test Method

1.1 This SOP is compliant with USEPA method 245.5, SW-846 method 7471A&B and CLP SOW ILM04.1.

2.0 Applicable Matrix or Matrices

2.1 This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.

3.0 Detection Limit

- 3.1 The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.
- 3.2 Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	SOLID/SOIL MDL/DL (mg/Kg)	SOLID/SOIL LOD (mg/Kg)	SOLID/SOIL ERL/LOQ (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Mercury	0.013	0.026	0.033	0.10	0.10

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	253.7

4.0 Scope of Application, Including Components to Be Analyzed

- 4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.
- 4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution procedure prior to analysis.
- 4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood.

These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

- 5.1 A weighed portion of the sample is acid digested for 2 minutes at $95\pm 2^{\circ}\text{C}$, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

6.0 Definitions

- 6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.
- 6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

- 7.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.
- 7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.
- 7.3 **Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.**
- 7.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

8.0 Safety

- 8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.
- 8.2 Normal accepted laboratory practices should be followed while performing this procedure.
- 8.3 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- 8.4 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

9.0 Equipment & Supplies

- 9.1 Perkin Elmer Flow Injection Mercury System (FIMS).
- 9.2 Perkin Elmer AS 90.
- 9.3 Mercury lamp.
- 9.4 Environmental Express Mod-Block digestion block capable of holding 95+2°C for 2 hours.
- 9.5 A scale or balance capable of weighing to 0.01 + 0.02 gram.
- 9.6 Snap cap digestion polypropylene vessels for use with the mod block digester. Five vessels of each lot must be taken through analysis to check for mercury.
- 9.7 Polypropylene watch glasses suitable for use with the above vessels in F above.
- 9.8 Manual Sample Mill
- 9.9 Wiley Sample Mill
- 9.10 Clippers for cutting vegetation

10.0 Reagents and Standards

- 10.0.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.0.2 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

10.1 REAGENTS

- 10.1.1 Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.
- 10.1.2 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃. Both HNO₃ and HCl must be of the reagent grade suitable for mercury determinations.
NOTE: This reagent is required for use when USACE project samples are being digested.
- 10.1.3 Concentrated HCl.
- 10.1.4 Concentrated HNO₃.
- 10.1.5 Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H₂O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.

- 10.1.6 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note: this is normally made up 2 liters at a time.
- 10.1.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.
- 10.1.8 3 % HCl carrier solution: 30 mLs HCl – 1 L DI H₂O; Prepare fresh daily.
- 10.1.9 Potassium persulfate 5% solution: Dissolve 100g in 2 liters of D.I. water. Used with digestion of CLP soils.

10.2 STANDARDS

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within Element as well as on the container's label.

10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.2.2 Preparation

10.2.2.1. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All Standards, blanks, and samples are taken through the digestion process.

10.2.2.2 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

10.2.2.3 Primary source and secondary source mercury standard solutions: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO₃ (200 ug/L).

10.2.3 Calibration standards:

Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.

10.2.3.1 Dilute the volumes below to 5 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 5 mLs for the initial step of the digestion.)

ug/L Hg

mLs of 200 ug/L standard in 50 mL

0.20	0.050
0.50	0.125
1.0	0.25
<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 50 mL</u>
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

10.2.3.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.3 Prepare one vessel of each.

10.2.3.4 It is necessary to digest the calibration standards when following all mercury methods.

10.2.4. Calibration verification standards:

10.2.4.1. Initial calibration verification (ICV) solution – 4.0 ug/L.

10.2.4.1.1 Prepared from the secondary source mercury standard (200 ug/L).

10.2.4.1.2 Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 It is necessary to digest the ICV standards when using all mercury methods for soil.

10.2.4.2 Continuing calibration verification (CCV) solution:

10.2.4.2.1 Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.

10.2.4.2.2 Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mLs with reagent water in a polypropylene digestion vessel.

10.2.4.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.2.4 It is necessary to digest the CCV standards when following all mercury methods for soil.

10.2.5 Digestion standards:

10.2.5.1 Laboratory control sample:

10.2.5.1.2 The Laboratory Control Sample (BS) is prepared from the secondary source mercury standard (200 ug/L) and added to ~ 0.3 grams of teflon chips.

10.2.5.1.3 Prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of teflon chips.

10.2.5.1.4 Appropriate reagents are added as below in the sample preparation section.

10.2.5.1.5 This solution is given a unique identifier in Element.

10.2.5.2 Matrix Spikes

10.2.5.2.1 Prepared from the primary or secondary source mercury standard (200 ug/L).

10.2.5.2.2 Prepared by adding 0.50 mL of the mercury standard (200 ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.

C10.2.5.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.

11.3 Refrigerate solid samples at 4°C ($\pm 2^\circ\text{C}$) upon receipt until digestion and analysis.

11.4 The sample should be analyzed without drying. A separate percent solids determination is required

11.5 The holding time for digestion of mercury samples is 28 days.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method.

Four BSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QUALITY CONTROL (Reference SW-846, 7471A Update III, 7471B Revision 2 February 2007, USEPA CLP ILMO 4.1 or EPA 245.5 for further clarification)

12.3.1 Daily

12.3.1.1 The instrument must be calibrated daily for all projects.

12.3.1.2 Begin each analysis with an ICB (concentration at or near mid range). The control limits are +10% for 7471A and 245.5, $\pm 20\%$ for 7471B and $\pm 5\%$ for 245.5..

12.3.1.3 Analyze ICB. Control limit is $< \pm \text{MDL}$ or $\pm \text{RL/CRDL}$ for other or CLP. For DOD, no analyte detected $> 2x \text{MDL}$.

- 12.3.1.4 If the ICV is not in control a new curve must be analyzed prior to sample analysis.
- 12.3.1.5 Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. The control limits are +20% for SW846-7471A, SW846 7471B and $\pm 10\%$ for 245.5. If an exceedance occurs, analyze another CCV, if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.
- 12.3.1.6 Follow each CCV with a CCB. Control limit is $< \pm \text{MDL}$ or $\pm \text{RL}/\text{CRDL}$ for others or CLP. For DOD, no analyte detected $> 2x \text{MDL}$.

12.3.2 Quarterly

- 12.3.2.1 IDLs for CLP (Follow SOP - 414).

12.3.3 Annually

- A. MDLs must be analyzed for all matrixes (Follow SOP - 414).

12.3.4 Digestion

- 12.3.4.1 BS data should be maintained and available for easy reference or inspection.

- 12.3.4.2 BLK ($< \pm \frac{1}{2} \text{RL}$ or $\pm \text{RL}$ for common contaminants or $\pm \text{RL}/\text{CRDL}$ for others or CLP)

- 12.3.4.2.1 Employ a minimum of one BLK per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be $< \pm \frac{1}{2} \text{RL}$ for USACE or $\pm \text{RL}/\text{CRDL}$ for others or CLP. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit or if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit, but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA officer will make this determination.

- 12.3.4.3 Laboratory control sample (BS).

- 12.3.4.3.1 Employ a minimum of one BS per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are +20% for SW846-7471A, 7471B and 245.5 solid samples. A BS will accompany each batch of soil samples. If the BS is not in control, the Inorganic Manager and QA Officer must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the

data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This corrective action is documented in a CAR.

12.3.5 Sample matrix

12.3.5.1 Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations.

12.3.5.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within +25% for 7471A and $\pm 20\%$ for 7471B of the true value (+20% for DOD projects). If results do not fall within the control limit redigestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your supervisor. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of + 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.4 For 245.5 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be 10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.3.5.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6

13.0 Calibration and Standardization

- 13.0.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.0.2 Set up the instrument with proper operating parameters.
- 13.0.3 Perkin Elmer Flow Injection Mercury System (FIMS).
- 13.0.3.1 Prepare the instrument for calibration by the following steps:
- 13.0.3.1.1 Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it, if it does replace it. (Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)
- 13.0.3.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
- 13.0.3.1.3 Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
- 13.0.3.1.4 Turn on computer and go to icon “AA Win LAB Analyst”
- 13.0.3.1.5 Go to method; select “Hg CAL 2” then OK.
- 13.0.3.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time = 18 sec.; BCC time = 2 sec.
- 13.0.3.1.7 Go to “Sample Info” and enter the order of the samples and other information that may be needed.
- 13.0.3.1.8 Save entered sample list under “Save ...sample info file” Note: description and batch ID are normally the date of analysis.
- 13.0.3.1.9 Go to “auto”; then to set-up. Select Browse in both spaces. One is to bring up your saved “Sample Information.” File. The other is to select a results library. Double click on heading and choose.
- 13.0.3.1.10 Turn the printer on.
- 13.0.3.1.11 Connect all tubing to the pump and blocks.
- 13.0.3.1.12 Start the pump by going to “FIAS” and click the pump 1 Icon (120).
- 13.0.3.1.13 The pump will start, then lock down and tighten the tubes onto the pump.
- 13.0.3.1.14 Turn on the nitrogen tank, it should be >500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.
- 13.0.3.1.15 The pressure gauge on the PE100 should be just below 100.
- 13.0.3.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.
- 13.0.3.1.17 Adjust the spring tension tubing until there is a constant “bubble of low rate” coming out to the waste tube.
- 13.0.3.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl₂. (click valve fill inject and make sure flow is correct and the line is rinsed)
- 13.0.3.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

13.0.3.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.

13.0.3.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".

13.0.3.1.22 "Select location" enter the #'s of the samples to be analyzed, then "OK".

13.0.3.2 Analyze the calibration standards as below.

13.0.3.2.1 A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within $\pm 10\%$ for SW846 7471A and $\pm 5\%$ for 245.5 methods, or $\pm 20\%$ for 7471B.

13.0.3.2.1 The curve should be linear with a calculated intercept with a minimum correlation coefficient of >0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

13.0.3.2.2 CLP requires a blank + 5 calibration standards (0, .02, .05, .1, .5 and $1.0\ \mu\text{g}$). (One standard must be at CRDL or IDL whichever is greater.)

14.0 Procedure

14.1 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.

14.2 Sample preparation:

14.2.1 It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

14.2.1.1 The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.

14.2.1.2 Two quarters should then be mixed to form halves.

14.2.1.3 The two halves should be mixed to form a homogenous matrix.

14.2.1.4 This procedure should be repeated several times until the sample is adequately mixed.

14.2.1.5 NOTE: Samples that are clay type materials must be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

14.2.2 Grinding of Vegetation Samples

14.2.2.1 Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to

- fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.
- 14.2.2.2 Transfer 0.30 g (for USACE work use anywhere from 0.20 to 1.0 g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and on the Element Batch Sheet. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. (Record in specific balance calibration log.)
 - 14.2.2.3 Add 2.5 mLs of reagent water, and 2.5 mLs of aqua regia and mix for samples. Add 2.5 mLs of aqua regia to standards and mix.
 - 14.2.2.4 Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at $95 \pm 2^\circ\text{C}$ (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature).
 - 14.2.2.5 Cool, bring to 30 ml with D.I. water.
 - 14.2.2.6 Add 7.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mLs).
- NOTE: The same amount of KMnO_4 added to the samples should be present in the standards and blanks.
- 14.2.2.7 Heat for 30 minutes on the hot block at $95 \pm 2^\circ\text{C}$ (The temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool. Samples may be saved at this point if there is not time to run the whole set that day.
 - 14.2.2.8 Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.
 - 14.2.2.9 Bring to 50 mLs with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. The samples are now ready for analysis.
 - 14.2.2.10 NOTE: Stannous Chloride (10.1.5) and 3% HCl (10.1.8) are added by the instrument during analysis.

14.2.3 Sample analysis

- 14.2.3.1 Set up the instrument as described in the calibration section above.
- 14.2.3.2 When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step "3". NOTE: When aqua-regia is added assume that all samples and standards have chlorine and treat accordingly. Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The

pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.

- 14.2.3.3 Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits of +10% for SW846-7471A or $\pm 20\%$ for 7471B and $\pm 5\%$ for 245.5 methods.
- 14.2.3.4 The ICB must follow the calibration standards ($< \pm \text{MDL}$ (USACE) or $\pm \text{RL/CRDL}$ for other or CLP), but not before the ICV. No analyte must be detected $> 2x\text{MDL}$ for DOD.
- 14.2.3.5 Each set of ten samples must be followed by a CCV with control limits of +20% for SW846-7471A and B and $\pm 10\%$ for 245.5 method. The run must also end with a CCV, then CCB.
- 14.2.3.6 Analyze CCB after calibration and each CCV. The CCB frequency is 10% or every 2 hours whichever is more frequent. (control limit is $< \pm \text{MDL}$ or $\pm \text{RL/CRDL}$ for other or CLP). For DOD, CCB at beginning and end of sequence and after every 10 samples. No analyte detected $> 2x\text{MDL}$.

14.2.3.7 Instrument Run Log example:

<u>AS LOC</u>	<u>Sample ID</u>
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ- ICV
10	SEQ-ICB
11	BS
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.2.3.8 Sample analysis:

14.2.3.8.1 Go to “Analyze”, “select location” and type in the range of numbers needed to complete analysis. (ie. 9-54). Press enter and the autosampler will proceed to enter the selected range.
NOTE: Check standards are loaded as part of the tray.

14.2.3.8.2 Make sure that the sample wash beaker is filled with 3% HCl.

14.2.3.8.3 Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are

made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL are reported flagged as estimated, (“B” flag).

14.2.4 Data reporting

14.2.4.1 Reduce data to result which will be reported using the soil spreadsheet found on the network..

14.2.4.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Pull up the blank spreadsheet at V: lab\metals\tests\mercury and transfer all the information pertinent to the current analysis. Save as the date of analysis. This information can be obtained from your mercury batch sheet.

15.3 Transfer the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

15.4 Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

15.5 The spreadsheet should divide the result which is the $\mu\text{g Hg}$ obtained from the sample mass by the sample mass in grams. This will yield a result of $\mu\text{g Hg/g}$ sample on a wet weight basis. Calculations in the spreadsheet should be checked occasionally to make sure that they are working correctly.

15.6 If available, divide the result by the %solids to obtain the result on a dry weight basis.

15.7 Report the data as $\mu\text{g Hg/g}$ of sample (mg/kg wet or mg/kg dry when % solids are available).

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.5 mL of the second source standard (200 ug/L) add to ~0.3g to 5 mLs with reagent water/aqua-regia in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS’s for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

17.0 Pollution Prevention

14.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

14.2 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

19.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

19.2.1 ICV not within + 10% (SW846) and (245.5)

19.2.1.1 If the problem is with the solution, re-prepare, obtain new stock if necessary.

19.2.1.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within + 20% (SW846) and (245.5)

19.2.2.1 If the problem is with the solution, reprepare, obtain new stock if necessary.

19.2.2.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

19.2.2.2.1 If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.

19.2.2.2.2 If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 CORRECTIVE ACTION: DIGESTION RELATED

19.3.1 The preparation blank less than $\pm \frac{1}{2}$ RL for DOD or \pm RL/CRDL for others or CLP.

19.3.1.1. If the problem is with the instrument or stannous chloride.

19.3.1.1.1 Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.

19.3.1.1.2 If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.

19.3.1.2 If the problem is with the digestion, all associated samples which are below the method detection limit (MDL) or have a level of mercury

greater than 10X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

19.3.2 BS not within control limits.

19.3.2.1 If the problem is with the instrument, reanalyze when instrument is in control with another aliquot of the sample.

19.3.2.2 If the problem is with the digestion.

19.3.2.2.1 If biased low, associated samples must be redigested.

19.3.2.2.2 If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

19.4 **CORRECTIVE ACTION: SAMPLE MATRIX RELATED**

19.4.1 Replicate analysis RPD not within +20%

19.4.1.1 The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within +25% 7471A and ±20% 7471B (+20% for DOD projects)

19.4.2.1 If the analyte level in the sample is greater than 4X the spiking level, the % recovery can not be evaluated and no action is taken.

19.4.2.2 If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III/IV); Method 7471A, 7471B*

21.2 *USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 245.1; APX-B*

21.3 *USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

- 22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

Addendum for USEPA CLP ILM 05.2

1. CCV concentration must be different from ICV.
2. The same CCV shall be used throughout analysis for a sample delivery group.
3. Calibration standards must be within 5% of the standard concentration.
4. 0.2 grams of sample must be used for the sample aliquot, add enough reagent water to each sample to make a total volume of 10 mL. Proceed with method as in the water method SOP 103.0 Revision 9.
5. The ICV and CCV must be at $\pm 20\%$ recovery.
6. A CRA must be analyzed at the beginning and end of each batch of 20 samples. Right after the ICV/ICB and right before the final CCV/CCB. The control limit is $\pm 30\%$.
7. The matrix spike must be analyzed at the concentration of 0.5 mg/Kg.

Table 1

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	SOLID/SOIL MDL/DL (mg/Kg)	SOLID/SOIL LOD (mg/Kg)	SOLID/SOIL ERL/LOQ (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Mercury	0.013	0.026	0.033	0.10	0.10

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	<ul style="list-style-type: none"> Daily ICAL prior to sample analysis Low standard at the RL/LOD level 	<ul style="list-style-type: none"> If more than one calibration standard is used, $r \geq 0.995$ Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-run curve Check instrument for maintenance needs <p>Samples cannot be analyzed until there is a passing calibration</p>
ICV	Alternate source standard to be analyzed after every calibration curve	Must be within $\pm 10\%$ for SW846 7471A, $\pm 20\%$ for 7471B, or $\pm 5\%$ for 245.5 of true value	<ul style="list-style-type: none"> Re-run ICV Repeat ICAL
CCV	<ul style="list-style-type: none"> After every 10 field samples and at the end of analysis sequence. 	<ul style="list-style-type: none"> $\pm 20\%$ for SW846-7471A&B, $\pm 10\%$ for 245.5 of true value 	<ul style="list-style-type: none"> Follow guidelines for SOP QS05
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> $\pm 20\%$ for SW846-7471A&B, $\pm 10\%$ for 245.5 of true value 	<ul style="list-style-type: none"> Follow guidelines for SOP QS05
BLK	One per prep 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 $> RL$	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Notify the PM for further action Re-prep of samples associated with the BLK NCR will be required for data reported
BS	One per prep batch	Most stringent criteria listed within the LIMS.	<ul style="list-style-type: none"> Re-analyze to confirm failed. Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. NCR will be required for data reported Follow guidelines from SOP QS05
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected $> LOD$.	<ul style="list-style-type: none"> Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Average percent recovery should be between 80-120%, with a 20% standard deviation. 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis
MDL Study	Once per year	<ul style="list-style-type: none"> • Calculated value must be less than the Spike level • 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	<ul style="list-style-type: none"> • Parameter must be detected • the response must be 3-times the noise level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	<ul style="list-style-type: none"> • Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: SW846 7471A/B, EPA245.5 (Mercury)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did blank spike(BS) meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				

- 11. Analytical results are correct and complete. _____
- 12. The appropriate SOP's have been used and followed. _____
- 14. "Raw data" including all manual integration's have been correctly interpreted. _____
- 15. "Special" sample preparation and analytical requirements have been met. _____
- 16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete. _____

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

1.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 105 REVISION #: 16 EFFECTIVE DATE: 041110

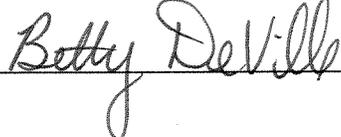
**METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE**

**References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3
February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B;
1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2**

APPROVALS:

Lab Director:  Date: 4/12/10

Data Quality Manager:  Date: 4/11/10

Section Supervisor:  Date: 4/13/10

Changes Summary

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *ITEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments

utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

- 6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.
- 6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1 Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

- 7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods

using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The

interference effects must be evaluated for each individual instrument since the intensities will vary.

7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.

7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.8 When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at \pm one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs.

Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized

7.4 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.

8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.

8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.

8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

- 9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- 9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- 9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.
- 9.4. Auto-sampler: Thermo Fisher or equivalent.
- 9.5. Printer capable of printing results every 4 minutes.
- 9.6. Cooling Water recycler.
- 9.7. Iteva software.
- 9.8. Argon gas supply – Liquid Argon
- 9.9. Class A volumetric flasks
- 9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001 gm).
- 9.11. Variable Eppendorf Pipettes 1000 μ L; 5000 μ L
- 9.12. Disposable beakers 10, 20 and 50 mL size.
- 9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

- 10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
- 10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.4. Nitric acid (concentrated), HNO₃. A method blank is digested and analyzed before a new lot number of HNO₃ is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.5. Calibration standards

10.5.1. All standards have an acid matrix of 2% HNO₃ and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).

10.5.2. CAL1 is the calibration blank: Reagent grade water **matrix matched as in 10.5.1. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**

10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.

10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

- 10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.
- 10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.

10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO₃ and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.

10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500

ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO₃ are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low

level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1 gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:

2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:

0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. The appropriate SOPs should be consulted regarding sample preparation. The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO₃ prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods 19th Edition Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO₃, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.1. Daily run and batch QC

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within $\pm 5\%$ for 200.7 and within $\pm 10\%$ for all other methods.

12.1.4. ICB/CCB less than two times \pm MDL or less than \pm LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within $\pm 20\%$ initially and client specific requirement of $\pm 30\%$ at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times \pm MDL or less than \pm LOD unless verified standard contamination for DOD. The IFB must recover within \pm 20% for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an "Q" on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within \pm 10% or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times \pm MDL or $<\pm$ LOD for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within \pm 10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75 to 125% for 6010B or 80 to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within \pm 10% of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

12.3.1. All quality control data should be maintained and available for easy reference or inspection.

12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than $\pm \frac{1}{2}$ RL for DOD or \pm RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.

12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are $\pm 15\%$ method 200.7 - aqueous and soil samples or $\pm 20\%$ for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are $>5x$ RL) or \pm the RL (if either are $<5x$ RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.

12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project

specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within $\pm 20\%$ of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply “J” flag for DOD if acceptance criteria are not met. Apply “N” flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

- 13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- 13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
- 13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO₃ / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.
- 13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ($\pm 10\%$) for 200.7 ($\pm 5\%$) and ICB ($< \pm 2 \times \text{MDL}$, $< \pm \text{LOD-DOD}$ or $\pm \text{RL/CRDL}$ for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within $\pm 20\%$ for DOD projects and $\pm 30\%$ for samples analyzed for 6010C. Then reanalyze the

highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: $< \pm\text{MDL}$ or $\pm\text{RL}/\text{CRDL}$ for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within $\pm 20\%$. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes $< \pm 2 \times \text{MDL}$ or $< \pm \text{LOD}$ for DOD beginning of sequence. Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be $< \text{LOD}$ (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within $\pm 20\%$ of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

- 1) Cal Std 1 (blank)
- 2) Cal Std 2 (Low Cal)
- 3) Cal Std 3 (Mid Cal)
- 4) Cal Std 4 (Ba @ 5000 ppb)
- 5) Cal Std 5 (QC5)
- 6) Cal Std 6 (QC 21)
- 7) Cal Std 7 (NAK 100)
- 8) Cal Std 8 (QC3)
- 9) Cal Std 9 (Ag)
- 10) Al IEC-(correction using ITEVA software)
- 11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

- 1) SEQ-ICV
- 2) SEQ-ICB
- 3) SEQ-CRL1-reporting limit standard 1
- 4) SEQ-CRL2-reporting limit standard 2
- 5) Ba@ 5000 ppb (readback)
- 6) QC5
- 7) NAK High-(readback)
- 8) QC 21 High-(readback)
- 9) Salt Cal at 500 ppm (readback)
- 10) Rinse
- 11) SEQ-IFA1
- 12) SEQ-IFB1
- 13) Rinse
- 14) SEQ-CCV
- 15) SEQ-CCB
- 16) Method Blank (*Batch # -BLK1*)
- 17) Blank Spike (*Batch # -BS1*)
- 18) Sample 1
- 19) Sample 2
- 20) Sample 3
- 21) Sample 4
- 22) Sample 5
- 23) Sample 6
- 24) Sample 7
- 25) Sample 8
- 26) Sample 9
- 27) Sample 10
- 28) SEQ-CCV
- 29) SEQ-CCB
- 30) Sample 11
- 31) Sample 12
- 32) Sample 13
- 33) Sample 14
- 34) Sample 15
- 35) Sample 16
- 36) Sample 17
- 37) Sample 18
- 38) Sample 19
- 39) Sample 20
- 40) Sample matrix spike (*batch#- MS1*)
- 41) Sample matrix spike duplicate (*batch# -MSD1*)
- 42) Sample post digestion spike (*batch# -PS1*)
- 43) Sample serial dilution (*batch# -DUP1*)
- 44) SEQ-CCV

- 45) SEQ-CCB
- 46) Preparation Blank (*batch# -BLK1*)
- 47) Blank Spike (*batch# -BS1*)
- 48) Sample 1
- 49) Sample 2
- 50) Sample 3
- 51) Sample 4
- 52) Sample 5
- 53) Sample 6
- 54) Sample 7
- 55) Sample 8
- 56) Sample 9
- 57) Sample10
- 58) SEQ-CCV
- 59) SEQ-CCB
- 60) Sample 11

RACK 2

- 1) Sample 12
- 2) Sample 13
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

- 14.1. Once the instrument has been calibrated, begin the analysis of samples.
- 14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**
- 14.3. Flush the system with 2% HNO₃ / 5% HCl for at least 1 minute before the analysis of each sample.
- 14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm 10\%$ of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**

14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within $\pm 10\%$ for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) $< 5\%$ from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be $< 2x \pm MDL$, for CLP $< RL$, for **DOD no analytes detected $> \pm LOD$** . If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank $< 1/10$ the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: $Ca \text{ mg/L} \times 2.5 + Mg \text{ mg/L} \times 4.1 = \text{total Hardness in mg/L as } CaCO_3$.

15.2. The instrument will generate data results in mg/L or $\mu\text{g/L}$ (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ (ug/g or mg/kg)} = IR * DF * FED / SM$$

SR	=	Sample result
IR	=	Instrument result ($\mu\text{g/L}$)
DF	=	Dilution factor (post digestion)
FED	=	Final volume of digestate (L)
SM	=	Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the supervisor for further processing and approval.

DOC LCS Preparation: See BS preparation under 10.7.1 through 10.7.3 above.

DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within $\pm 50\%$.

17. Pollution Prevention:

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

- 19.1.1. ICV not within $\pm 10\%$ or $\pm 5\%$ for 200.7
 - a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.

- b. Is the problem with the calibration?
 - i. Recalibrate through analysis of appropriate standards and recheck ICV.
- 19.1.2. ICB not \pm MDL or within \pm 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**
- a. Is the problem with the solution?
 - i. Re-prepare
 - b. Is the problem with the calibration?
 - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.
- 19.1.3. Check standards not within \pm 5%
- a. Is the problem with the solution?
 - i. Re-pour, re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.4. CLP only-CRI not within \pm 20% (Internal QC, only required for CLP work).
- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes $<\pm$ LOD.**
- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.6. IFB not within \pm 20%
- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.7. CCV not within \pm 10%
- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

- 19.1.8.. CCB not $\pm 2 \times$ MDL or CRDL for CLP, DOD no analytes detected $> \pm$ LOD.
- a. Is the problem with the solution?
 - i. Re-prepare
 - b. Is the problem with the calibration?
 - i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

- 19.2.1. Preparation blank (BLK) not within $\pm \frac{1}{2}$ RL and \pm RL for common contaminants DOD or RL/CRDL for other or CLP
- a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.
- 19.2.2. BS not within control limits
- a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If biased low, associated samples must be re-digested.
 - ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

- 19.3.1. Replicate analysis RPD not within $\pm 20\%$ (if both are $> 5X$ CRDL) or \pm the CRDL (if either are $< 5X$ CRDL).
- a. The associated sample data must be qualified on the final report.
- 19.3.2. Spike analysis recovery not within $\pm 20\%$.
- a. Is the analyte level in the sample greater than 4X the spiking level?
 - i. If yes, the spike recovery is not evaluated.
 - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.
- 19.3.3. When required, post digestion spike analysis recovery not within $\pm 25\%$ for SW6010B, DOD or $\pm 20\%$ SW6010C.
- a. The associated sample data must be qualified on the final report.
 - b. For USACE analysis by MSA is required.
- 19.3.4. Serial dilution analysis percent difference not within $\pm 10\%$
- a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

- i. If no, the serial dilution data can not be evaluated.
- iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B and Method 6010C.*

21.2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 200.7; APX-B.*

21.3. *USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.1. (Based on NELAC Voted Revision June 5, 2003. 4/22/09

22. Tables, Diagrams, Flowcharts and Validation Data

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L
Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Interference Check	<ul style="list-style-type: none"> once per calibration 	<ul style="list-style-type: none"> IFA less than LOD if not verified contamination of standard. IFB must be within $\pm 20\%$. 	<ul style="list-style-type: none"> Check IEC corrections for metals in the IFA.
Calibration Curve	<ul style="list-style-type: none"> Prior to analyzing any samples A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C). 	<ul style="list-style-type: none"> Linear calibration Corr. of 0.998 Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICB	At the beginning of every sequence	Must meet the $< \pm \text{LOD}$ for DOD or $< 2 \times \text{MDL}$	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-client samples 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than $\frac{1}{2} \pm \text{RL}$. 	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action Re-prepare of samples associated with the MB NCR will be required for data reported Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	<ul style="list-style-type: none"> • Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Must meet the criteria of the BS for average accuracy 	<ul style="list-style-type: none"> • Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):				
Batch Number(s):				
Method: 6010B or 6010C (ICP)				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did BS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____ Date: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

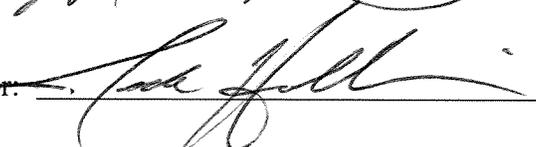
ORGANICS: SOP 201 REVISION #: 20 EFFECTIVE DATE: 042610

**GC/MS SEMIVOLATILES and LOW-CONCENTRATION PAHs
BY EPA METHOD 625 AND SW846 METHOD 8270C AND 8270D
INCLUDING ADDITIONAL APPENDIX IX COMPOUNDS**

APPROVALS:

Lab Director:  Date: 4/27/10

Data Quality Manager:  Date: 4/27/10

Section Supervisor:  Date: 4/27/10

Changes Summary

Revision 20, 4/13/10

- The SOP is an update from Revision 19 dated 4/11/2010
- The SOP is formatted to simplify the text and place all method/program specifications in the SOP tables.

Revision 19, 4/11/10

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

Table of Contents

1. Identification of the Test Method
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18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
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1.0 Identification of the Test Method

This SOP is based primarily on SW-846 Methods 8000B/8000C/8270C/8270D. Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This SOP is used for the analysis of semi-volatile organic compounds (including low concentration PAHs) in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits – Reporting Limits

See Table 1

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is routinely analyzed and reported under the scope of this SOP is listed in the Appendix of this SOP. This table also lists the associated Detection Limit, Limit of Detection and Reporting Limit (also defined as the Limit of Quantitation).

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

6.0 Definitions –

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.

- 8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
- 8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 8.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

9.0 Equipment & Supplies

- a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for split-less injection.
- b Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column.
- c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.
- d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.
- e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.
- f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- g Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified times or scan-number limits.
- h Micro syringes – gas tight 5µL and larger.
- i Liners – 2mm or 4mm single goose-neck.
- j Septa 11mm.
- k Seals- dual vespel stainless steel or gold plated 0.8mm.
- l Vials- 2ml and larger amber.
- m Volumetric flasks- 10ml and larger class A with glass stopper.

10.0 Reagents and Standards –

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory.
- 10.2 Reagent grade chemicals shall be used in all tests unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 10.3 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.
- 10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature are monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook.
- 10.5 Individual standard makeup is recorded in LIMS with specific details concerning the standard being used, concentration, amount, solvent and expiration date.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C . All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C . Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

- 12.1 Internals - All samples and QC are spiked with internal standards prior to analysis.
- 12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.
- 12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.
- 12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.
- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:
 - 12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
 - 12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

- 12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.
- 12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.
- 12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Initial Calibration - An initial multi-point calibration curve must be analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve range from 1.0ug/mL to 100ug/mL for regular SVOCs and 0.1µg/mL to 50µg/mL for low-concentration PAHs.. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 13.3 Initial Calibration Verification (ICV) - A second source standard at the continuing calibration verification (CCV) level must be analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 13.4 Continuing Calibration Verification (CCV) - Every 12 hours, a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.0 Procedure

Prior to analysis the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, 3546 3550, 3580, EPA method 625 or CLP).

- 14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.
- 14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard. The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port . See **Table 2** for criteria and corrective action.
- 14.3 Extracts - Prior to analysis, 1.0 mL extracts are prepared by verifying volume and spiking with 20uL of the internal standard solution.

14.5 Instrument sequence-The instrument sequence log is filled out prior to sample analyses. An example of a typical instrument sequence log follows:

- 1-SEQ-TUN1 (12:00 am)
- 2-SEQ-CCV1
- 3-SEQ-BS1
- 4-SEQ-BLK1
- 5-Sample
- 6-Sample
- 7-Sample
- 8-Sample
- 9-Sample
- 10-Sample
- 11-Sample
- 12-Sample
- 13-Sample
- 14-SEQ-MS1
- 15-SEQ-MSD1
- 16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
- 17-SEQ-CCV2
- 18-Sample
- 19-Sample
- 20-Sample

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need reanalysis or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Internal Standard Area Counts and Retention Times

14.6.2 Surrogate Recoveries and Retention Times

- 14.6.3 Analyte concentration.
- 14.6.4 Analyte identification based on spectrum and retention time.
- 14.6.5 Analyte quantitation verification.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where:

Calculated concentration is determined from the initial calibration.

Theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where:

CCV RF is the response factor from the analysis of the verification standard

Average RF is the average of the response factors from the initial calibration.

15.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to $\mu\text{g/L}$.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume extracted in $\mu\text{g/L}$.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (μL). The nominal injection volume for samples and calibration standards must be the same.

- \overline{RF} = Mean response factor from the initial calibration.
 V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

- 15.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s ,

A_{is} , C_{is} , D , and \overline{RF} are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

- 15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, Technical Director and/or Data Quality Manager.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

- 16.1 Method Detection Limit Study or Detection Limit Determination
- 16.2 Limit of Detection Verification
- 16.3 Limit of Quantitation or Reporting Limit Verification
- 16.4 Demonstration of Capability (DOC)
- 16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

40 CFR, Part 136; Appendix A

Test Methods for Evaluating Solid Waste, SW-846

National Environmental Laboratory Accreditation Conference; CH. 5, 2003

USACE, EM 200-1-3; Appendix 1; Shell, 2/2001

DOD Quality Systems Manual for Environmental Laboratories,

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).

22.2 Table 2, QA/QC summary table

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist(s)

22.5 Table 5, 625 QC Limits

22.6 Table 6, Standards Used

22.7 Table 7, INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis

22.8 Table 8, LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

22.9 Figure 1, Tailing Factor Calculation

22.10 Table 9, DFTPP Tuning Criteria

TABLE 1

Analyte (Water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3'-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L

Table 1 (Continued)

Analyte (Water)	DL	LOD	MRL/LOQ	Units
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
Analyte (Soil)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3'-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg

Table 1 (Continued)

Analyte (Soil)	DL	LOD	MRL/LOQ	Units
Benzo (a) pyrene	83.3	167	333	ug/Kg
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg
Analyte Low PAH (Water)	DL	LOD	MRL/LOQ	Units
1-Methylnaphthalene	0.0500	0.100	0.200	ug/L
2-Methylnaphthalene	0.0500	0.100	0.200	ug/L
Acenaphthene	0.0500	0.100	0.200	ug/L
Acenaphthylene	0.0500	0.100	0.200	ug/L
Anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) pyrene	0.0500	0.100	0.200	ug/L
Benzo (b) fluoranthene	0.0500	0.100	0.200	ug/L
Benzo (g,h,i) perylene	0.0500	0.100	0.200	ug/L
Benzo (k) fluoranthene	0.0500	0.100	0.200	ug/L
Chrysene	0.0500	0.100	0.200	ug/L
Dibenz (a,h) anthracene	0.0500	0.100	0.200	ug/L
Fluoranthene	0.0500	0.100	0.200	ug/L
Fluorene	0.0500	0.100	0.200	ug/L
Indeno (1,2,3-cd) pyrene	0.0500	0.100	0.200	ug/L
Naphthalene	0.0500	0.100	0.200	ug/L
Phenanthrene	0.0500	0.100	0.200	ug/L
Pyrene	0.0500	0.100	0.200	ug/L
Analyte Low PAH (Soil)	DL	LOD	MRL/LOQ	Units
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg

Table 1 (Continued)

Analyte Low PAH (Soil)	DL	LOD	MRL/LOQ	Units
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg
Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
Analyte (TCLP)	DL	LOD	MRL/LOQ	Units
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270)

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 of DoD QSM 4.1).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C of DoD QSM 4.1. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 8 of this SOP.	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. [Method 625 – benzidine and pentachlorophenol tailing limits are 3 and 5, respectively, when benzidine or acids are target analytes. Benzidine tailing is specific to benzidine analysis and pentachlorophenol tailing is specific to acid analyte analyses according to 625.]	Correct problem then repeat breakdown checks.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq 20\%$. Not applied when low concentration PAHs are the only target analytes.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (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: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs</p> <p>2. RSD for RFs for CCCs: SVOCs $\leq 30\%$ and one option below: Option 1: RSD for each analyte $\leq 15\%$; [$\leq 20\%$ for non-DoD 8270D; or, $\leq 35\%$ for non-DoD 625] Option 2: linear least squares regression $r \geq 0.995$ or $r^2 \geq 0.990$; [$r \geq 0.990$ for non-DoD analyses] Option 3: non-linear regression—coefficient of determination (COD) $r^2 \geq 0.990$ (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 for DoD projects.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value [$\pm 25\%$ for non-DoD 8270C; or, $\pm 30\%$ for non-DoD 8270D]	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 should 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 sequence CCV is used.	NA.	NA.	

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (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. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs 2. %Difference/Drift for all target compounds and surrogates: SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ for CCCs only non-DoD 8270C]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data should be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8270C, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem should be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	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 qualifier 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.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL/LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL/LOQ.	Correct problem. 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 client or DoD (appendix G), if available. AFCEE 4.0.02 limits are applied for low concentration PAHs as they are not addressed by DoD. 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. Low concentration PAH limits	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.	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	Use LCS criteria, above.	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 qualifier 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	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: $RPD \leq 30\%$ or client specified limit (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 qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria			Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	Surrogate	Water	Solid	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 qualifier to all associated analytes if acceptance criteria are not met. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.	
		Nitrobenzene-d5	40-110	35-100			
		2-Fluorobiphenyl	50-110	45-105			
		Terphenyl-d14	50-135	30-125			
		Phenol-d6	10-115	40-100			
		2-Fluorophenol	20-110	35-105			
		2,4,6-Tribromophenol	40-125	35-125			
		QC acceptance criteria specified by DoD (above) or Client. Low PAH surrogate limits are 14%-129% soil and 34%-167% water. Otherwise, in-house control limits may be used. No limits specified for Method 625.					
Results reported between DL and LOQ	NA.	NA.			NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory or senior-scientist approval for manual integrations on standards and batch QC samples
7. Was the data uploaded into LIMS via direct upload (i.e. datatool) – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8260B/624/8270C/8270D/625 (Circle One)

QA/QC Item	Yes	No	NA	Second Review	Level
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?					
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).					
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?					
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.					
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?					
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?					
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?					
8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?					

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 - 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Table 6 - BNA STANDARDS USED

<u>base/neutral mix (2000ppm)</u>	<u>acids mix (2000ppm)</u>
bis(2-Chloroethyl)ether	2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether	2-Methylphenol
1,3-Dichlorobenzene	4-Methylphenol
1,2-Dichlorobenzene	Benzoic acid
1,4-Dichlorobenzene	4,6-Dinitro-2-methylphenol
Hexachloroethane	4-Nitrophenol
N-Nitroso-di-methylamine	2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine	2,4,6-Trichlorophenol
2,4-Dinitrotoluene	Phenol
2,6-Dinitrotoluene	Pentachlorophenol
Fluorene	2-Nitrophenol
Dimethylphthalate	4-Chloro-3-methylphenol
Hexachlorocyclopentadiene	2,4-Dichlorophenol
Anthracene	2,4-Dimethylphenol
4-Bromophenyl-phenylether	Benzoic acid
Di-n-butylphthalate	
bis(2-Chloroethoxy)methane	
1,2-Diphenylhydrazine	<u>semivoa misc. mix(2000ppm)</u>
Fluoranthene	Aniline
Hexachlorobenzene	Benzyl alcohol
N-Nitrosodiphenylamine	Carbazole
Phenanthrene	4-Chloroaniline
Hexachlorobutadiene	Dibenzofuran
Isophorone	2-Methylnaphthalene
Naphthalene	2-Nitroaniline
Nitrobenzene	3-Nitroaniline
1,2,4-Trichlorobenzene	4-Nitroaniline
Acenaphthene	Pyridine
Acenaphthylene	
2-Chloronaphthalene	<u>Benzidine mix (2000ppm)</u>
4-Chlorophenyl-phenylether	Benzidine
Diethylphthalate	3,3'-Dichlorobenzidine
Benzo(a)anthracene	
Bis(2-ethylhexyl)phthalate	
Butylbenzylphthalate	
Chrysene	<u>Individual or misc. mixes (2000/5000/20,000ppm)</u>
p-(Dimethylamino)azobenzene	Caprolactam
Pyrene	Benzaldehyde
Benzo(b)fluoranthene	Atrazine
Benzo(k)fluoranthene	1,1'-Biphenyl
Benzo(g,h,i)perylene	1,4-Dioxane
Benzo(a)pyrene	1-methylnaphthalene
Dibenz(a,h)anthracene	2,6-dichlorophenol
Di-n-octylphthalate	2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene	

<u>BNA internals (2000ppm)</u>	<u>Acid surrogate (7500ppm)</u>
1,4-Dichlorobenzene-d4 (L.S)(1)	2-Fluorophenol (S)
Naphthalene-d8 (L.S)(35)	Phenol-d6 (S)
Acenaphthene-d10 (L.S) (59)	2,4,6-Tribromophenol (S)
Phenanthrene-d10 (L.S) (79)	2,-Chlorophenol-d4 (S)
Chrysene-d12 (L.S) (92))	<u>BN surrogate (5000ppm)</u>
Perylene-d12 (L.S) (101)	Nitrobenzene-d5 (S)
	Terphenyl-d14 (S)
	2-Fluorobiphenyl (S)
	1,2-Dichlorobenzene-d4 (S)

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis					
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 (I.S) (79)		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 (I.S)(35)		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 (I.S) (92)		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 (I.S) (59)		164	Perylene-d12 (I.S) (101)		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276

I.S=internal standard, Q.M=quant mass, S=surrogate

**Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS –
Standard SVOC analysis (contd)**

COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
O-Toluidine	1	106		59	
	1		Phenanthrene-d10 (I.S) (79)		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 (I.S)(35)		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 (I.S) (92)		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 (I.S) (59)		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 (I.S) (101)	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	

I.S=internal standard, Q.M=quant mass, S=surrogate

Table 8: LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

INTERNAL STD ASSOCIATION

Phenanthrene-d10 (IS)

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene

2-Fluorobiphenyl(SUR)

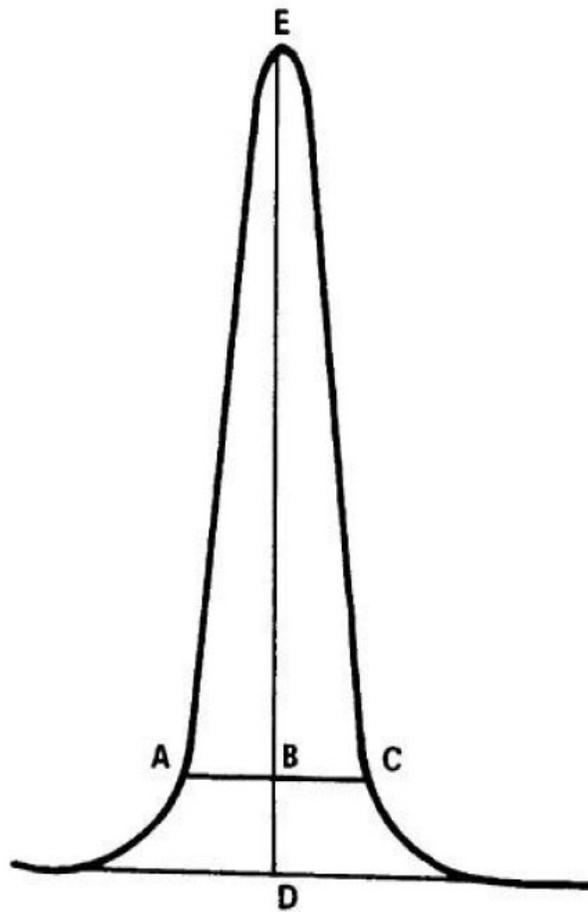
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene

Perylene-d12 (IS)

Terphenyl-d14(SUR)

Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

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

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Table 9, DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note: While 8270D table 3 indicates different criteria, section 11.3.1.2 allows the use of alternate criteria.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 202

REVISION #: 23

EFFECTIVE DATE: 20100909

**GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B
INCLUDING APPENDIX IX COMPOUNDS**

APPROVALS:

Lab Director:  _____ Date: 9/9/10

Data Quality Manager:  _____ Date: 9/9/10

Section Supervisor:  _____ Date: 9/9/10

Changes Summary

Revision 23, 09/09/10

- This SOP is an update from Revision 22 dated 09/30/09.
- Tables 1 and 2 have been updated with appropriate reference updates.
- Tables 5-7 have been added.

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

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1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. Detection Limit: See **Table 1** of this SOP.

4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. Definitions

6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. Safety

- 8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9. Equipment & Supplies

- 9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.
- 9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 μm film thickness or 20 meter x 0.18 mm ID 1.0 μm film thickness silicon coated fused silica capillary column or equivalent.
- 9.3 M.S.: HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 μL (50 ng) of the GC/MS tuning standard is introduced to the GC.]
- 9.4 Purge and Trap Unit
 - 9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.
 - 9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.
- 9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- 9.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.
- 9.7 Microsyringes – 1.0, 5.0, 10, 25, 100, 250, 500 and 1000 μL .
- 9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.
- 9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.
- 9.10 Disposable pasteur pipets.
- 9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.
- 9.12 Spatula - stainless steel.
- 9.13 Glass scintillation vials - 20mL with screw caps.
- 9.14 Nitrile Gloves
- 9.15 pH paper (measures pH from 0-14).

10. Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.2 Organic-free reagent water - obtained from the charcoal filter system in the VOA laboratory.
- 10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)
- 10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)
- 10.5 Sodium bisulfate, NaHSO₄ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- 10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ or less from the date they are received/prepared. The freezer temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.
- 10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50 μL syringe, 40 μL of standard (BFB @ 2500ng/ μL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same making a 50ng/ μL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ or less for up to 6 months (**1 week for South Carolina samples**). A direct injection of 1 μL (or equivalent purge) is used to tune the instrument.
- 10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor, Lot) and diluted to volume with same making a 150ng/ μL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1 μL of this standard. (The internal standard/surrogate solution may be replaced if the -50% - 200% criteria fails in the CCV when calculated against the previous CCV.)

Standard	Conc. (ng/μL)	Syringe (μL)	Amount (μL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the LIMS for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 1 week. A 100μg/L (5mL purge) standard is made using 50μL of this standard to 50mL of reagent water.

Stock Standard(CCV)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE (Cat#30265)	20000	25	20	200
Vinyl Acetate (#3766)	5000	100	80	200
Ketones (cat#30006)	5000	100	80	200
Liquid mix (C-349H-07)	2000	100	100	100
Custom mix (CCS-1037)	5000	50	40	100
Gases (cat#30042)	2000	100	100	100
Acrolein/Acrylonitrile (CC2098.10)	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 1 week. A 50μg/L ICV/LCS/Matrix Spike is made using 25μL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

11. Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 All water samples are stored in the “True” refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). The temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. The weekend temperature is monitored with a Min/Max thermometer and recorded upon arrival next business day.

12. Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for acceptance criteria and corrective action.
- 12.2 Surrogates - All samples and QC are spiked with surrogates. See **Table 2** of this SOP for acceptance criteria and corrective action.
- 12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 μg/L when analyzing 624 samples (QC Check Sample). See **Table 2** of this SOP for acceptance criteria and corrective action. **When analyzing samples for South Carolina the limits are 70-130% except for poor purgers which are 60-140%.**
- 12.4 Method Blanks - A method blank is analyzed every 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action..
- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. See **Table 2** of this SOP for acceptance

criteria and corrective action. MS data evaluation must include the consideration of the following factors.

- 12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
- 12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.
- 12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13. Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.
- 13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0 μ L injection of the tuning standard. See **Table 5** of this SOP for acceptance criteria. Tune must be met every 12 hours sample analysis is to be performed (**every 24 hours for *Federal Register Method 624* except for South Carolina which only allows 12 hours**). The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with background subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

- 13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. See the LIMS for preparation of standards. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate**

solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate. All manual calibration integrations must be approved by the section manager or designated peer reviewer.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** See **Table 2** of this SOP for acceptance criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual calibration integrations must be approved by the section manager or designated peer reviewer Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride
SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

13.5.2 Initial Calibration Verification (ICV) - A second source standard is prepared at or near the CCV concentration and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual ICV integrations must be approved by the section manager or designated peer reviewer.

13.5.3 Continuing Calibration Verification (CCV) - A CCV is analyzed every 12 hour tune and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual CCV integrations must be approved by the section manager or designated peer reviewer. .

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check

sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

14. Procedure

14.1 LCS - An LCS is analyzed every 12 hour tune. Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See **Table 2** of this SOP for acceptance criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample).**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a non-conformance report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 Instrument sequence

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)

2-CCV

3-LCS

4-Method Blank

5-Sample

6-Sample

7-Sample

8-Sample

9-Sample

10-Sample

11-Sample

12-Sample

13-Sample

14-Sample

- 15-Sample
- 16-Sample
- 17-Sample MS
- 18-Sample MSD
- 19-BFB (12:00pm - 12 hours since last BFB/CCV)
- 20-CCV
- 21-LCS
- 22-Method Blank
- 23-Sample
- 24-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. See **Table 2** of this SOP for acceptance criteria and corrective action. Formal data evaluation is detailed in SOP QS05. See **SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas counts and retention times.

14.5.2 Surrogates – Recoveries and retention times.

Federal Register Method 624 contains no criteria for surrogate recovery.

Surrogate	WATER	SOIL
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration.

14.5.4 Qualitative identification based on spectrum and retention time.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

- 15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within $\pm 20\%$ for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

- 15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, $D = 1$. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{\text{RF}}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

- 15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(W_s)(1000)}$$

where: A_s , A_{is} , C_{is} , D , and \overline{RF} are the same as for aqueous samples.
 W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See [Table 2](#) for acceptance criteria.

17. Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. [Table 2](#) of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. [Table 2](#) within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. Waste Management.

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

- 21.1 40 CFR, Part 136; Appendix A
- 21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
- 21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
- 21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
- 21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005

22. Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).
- 22.2 Table 2, QA/QC summary table
- 22.3 Table 3, Technical Completeness / Accuracy Checklist
- 22.4 Table 4, Data Reviewers Checklist(s)
- 22.5 Table 5, BFB Tuning Criteria
- 22.6 Table 6, Analyst Checklist
- 22.7 Table 7, INTERNAL STANDARD ASSOCIATION

Table 1 – DL/LOD/LOQ

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,1,1,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,1-Trichloroethane (1,1,1-TCA)	1.25	2.50	5.00	ug/Kg
1,1,2,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	2.50	5.00	10.0	ug/Kg
1,1,2-Trichloroethane	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethane (1,1-DCA)	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethene (1,1-DCE)	1.25	2.50	5.00	ug/Kg
1,1-Dichloropropene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichloropropane	1.25	2.50	5.00	ug/Kg
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,4-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,2-Dibromo-3-chloropropane (DBCP)	2.50	5.00	10.0	ug/Kg
1,2-Dibromoethane (EDB)	1.25	2.50	5.00	ug/Kg
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2-Dichloroethane (EDC)	1.25	2.50	5.00	ug/Kg
1,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,3,5-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
2,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/Kg
2-Chlorotoluene	1.25	2.50	5.00	ug/Kg
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/Kg
4-Chlorotoluene	1.25	2.50	5.00	ug/Kg
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/Kg
Acetone	5.00	10.0	20.0	ug/Kg
Acrolein	5.00	10.0	20.0	ug/Kg
Acrylonitrile	5.00	10.0	20.0	ug/Kg
Benzene	1.25	2.50	5.00	ug/Kg
Bromobenzene	1.25	2.50	5.00	ug/Kg
Bromochloromethane	1.25	2.50	5.00	ug/Kg
Bromodichloromethane	1.25	2.50	5.00	ug/Kg
Bromoform	1.25	2.50	5.00	ug/Kg
Bromomethane	2.50	5.00	10.0	ug/Kg
Carbon Disulfide	1.25	2.50	5.00	ug/Kg
Carbon Tetrachloride	1.25	2.50	5.00	ug/Kg
Chlorobenzene	1.25	2.50	5.00	ug/Kg
Chloroethane	2.50	5.00	10.0	ug/Kg
Chloroform	1.25	2.50	5.00	ug/Kg
Chloromethane	2.50	5.00	10.0	ug/Kg
cis-1,2-Dichloroethene (cis-1,2-DCE)	1.25	2.50	5.00	ug/Kg
cis-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Cyclohexane	1.25	2.50	5.00	ug/Kg
Dibromochloromethane	1.25	2.50	5.00	ug/Kg

Analyte	MDL/DL	LOD	MRL/LOQ	Units
Dibromomethane	1.25	2.50	5.00	ug/Kg
Dichlorodifluoromethane (CFC-12)	2.50	5.00	10.0	ug/Kg
Ethyl methacrylate	1.25	2.50	5.00	ug/Kg
Ethylbenzene	1.25	2.50	5.00	ug/Kg
Hexachlorobutadiene	1.25	2.50	5.00	ug/Kg
Iodomethane	5.00	10.0	20.0	ug/Kg
Isopropylbenzene (Cumene)	1.25	2.50	5.00	ug/Kg
Methyl Acetate	2.50	5.00	10.0	ug/Kg
Methyl methacrylate	1.25	2.50	5.00	ug/Kg
Methyl Tertiary Butyl Ether (MTBE)	1.25	2.50	5.00	ug/Kg
Methylcyclohexane	1.25	2.50	5.00	ug/Kg
Methylene Chloride, or Dichloromethane	2.50	5.00	10.0	ug/Kg
Naphthalene	1.25	2.50	5.00	ug/Kg
n-Butylbenzene	1.25	2.50	5.00	ug/Kg
n-Propylbenzene	1.25	2.50	5.00	ug/Kg
p-Isopropyltoluene	1.25	2.50	5.00	ug/Kg
sec-Butylbenzene	1.25	2.50	5.00	ug/Kg
Styrene	1.25	2.50	5.00	ug/Kg
tert-Butylbenzene	1.25	2.50	5.00	ug/Kg
Tetrachloroethene (PCE; PERC)	1.25	2.50	5.00	ug/Kg
Toluene	1.25	2.50	5.00	ug/Kg
trans-1,2-Dichloroethene (trans-1,2-DCE)	1.25	2.50	5.00	ug/Kg
trans-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Trichloroethene (TCE)	1.25	2.50	5.00	ug/Kg
Trichlorofluoromethane (CFC-11)	2.50	5.00	10.0	ug/Kg
Vinyl acetate	2.50	5.00	10.0	ug/Kg
Vinyl Chloride (VC)	2.50	5.00	10.0	ug/Kg
m,p-Xylene	2.50	5.00	10.0	ug/Kg
o-Xylene	1.25	2.50	5.00	ug/Kg
1,1,1,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,1-Trichloroethane (1,1,1-TCA)	0.25	0.50	1.00	ug/L
1,1,2,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	0.50	1.00	2.00	ug/L
1,1,2-Trichloroethane	0.25	0.50	1.00	ug/L
1,1-Dichloroethane (1,1-DCA)	0.25	0.50	1.00	ug/L
1,1-Dichloroethene (1,1-DCE)	0.25	0.50	1.00	ug/L
1,1-Dichloropropene	0.25	0.50	1.00	ug/L
1,2,3-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,3-Trichloropropane	0.50	1.00	2.00	ug/L
1,2,4-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,4-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,2-Dibromo-3-chloropropane (DBCP)	0.50	1.00	2.00	ug/L
1,2-Dibromoethane (EDB)	0.25	0.50	1.00	ug/L
1,2-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,2-Dichloroethane (EDC)	0.25	0.50	1.00	ug/L
1,2-Dichloropropane	0.25	0.50	1.00	ug/L
1,3,5-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,3-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,3-Dichloropropane	0.25	0.50	1.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,4-Dichlorobenzene	0.25	0.50	1.00	ug/L
1-Chlorohexane	0.50	1.00	2.00	ug/L
2,2-Dichloropropane	0.25	0.50	1.00	ug/L
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/L
2-Chloroethyl vinyl ether	1.25	2.50	5.00	ug/L
2-Chlorotoluene	0.25	0.50	1.00	ug/L
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/L
4-Chlorotoluene	0.25	0.50	1.00	ug/L
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/L
Acetone	2.50	5.00	10.0	ug/L
Acrolein	1.25	2.50	5.00	ug/L
Acrylonitrile	2.50	5.00	10.0	ug/L
Benzene	0.25	0.50	1.00	ug/L
Bromobenzene	0.25	0.50	1.00	ug/L
Bromochloromethane	0.25	0.50	1.00	ug/L
Bromodichloromethane	0.25	0.50	1.00	ug/L
Bromoform	0.25	0.50	1.00	ug/L
Bromomethane	0.50	1.00	2.00	ug/L
Carbon Disulfide	0.25	0.50	1.00	ug/L
Carbon Tetrachloride	0.25	0.50	1.00	ug/L
Chlorobenzene	0.25	0.50	1.00	ug/L
Chloroethane	0.50	1.00	2.00	ug/L
Chloroform	0.25	0.50	1.00	ug/L
Chloromethane	0.25	0.50	1.00	ug/L
cis-1,2-Dichloroethene (cis-1,2-DCE)	0.25	0.50	1.00	ug/L
cis-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Cyclohexane	0.25	0.50	1.00	ug/L
Dibromochloromethane	0.25	0.50	1.00	ug/L
Dibromomethane	0.25	0.50	1.00	ug/L
Dichlorodifluoromethane (CFC-12)	0.50	1.00	2.00	ug/L
Di-isopropyl ether	0.25	0.50	1.00	ug/L
ETBE	0.25	0.50	1.00	ug/L
Ethyl methacrylate	0.25	0.50	1.00	ug/L
Ethylbenzene	0.25	0.50	1.00	ug/L
Hexachlorobutadiene	0.25	0.50	1.00	ug/L
Iodomethane	0.25	0.50	1.00	ug/L
Isopropylbenzene (Cumene)	0.25	0.50	1.00	ug/L
Methyl Acetate	0.50	1.00	2.00	ug/L
Methyl methacrylate	0.25	0.50	1.00	ug/L
Methyl Tertiary Butyl Ether (MTBE)	0.25	0.50	1.00	ug/L
Methylcyclohexane	0.25	0.50	1.00	ug/L
Methylene Chloride, or Dichloromethane	0.50	1.00	2.00	ug/L
Naphthalene	0.25	0.50	1.00	ug/L
n-Butylbenzene	0.25	0.50	1.00	ug/L
n-Propylbenzene	0.25	0.50	1.00	ug/L
p-Isopropyltoluene	0.25	0.50	1.00	ug/L
sec-Butylbenzene	0.25	0.50	1.00	ug/L
Styrene	0.25	0.50	1.00	ug/L
t-Butyl alcohol	1.25	2.50	5.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
tert-Amyl methyl ether	2.50	5.00	10.0	ug/L
tert-Butylbenzene	0.25	0.50	1.00	ug/L
Tetrachloroethene (PCE; PERC)	0.25	0.50	1.00	ug/L
Tetrahydrofuran	1.25	2.50	5.00	ug/L
Toluene	0.25	0.50	1.00	ug/L
trans-1,2-Dichloroethene (trans-1,2-DCE)	0.25	0.50	1.00	ug/L
trans-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Trichloroethene (TCE)	0.25	0.50	1.00	ug/L
Trichlorofluoromethane (CFC-11)	0.50	1.00	2.00	ug/L
Vinyl acetate	1.25	2.50	5.00	ug/L
Vinyl Chloride (VC)	0.50	1.00	2.00	ug/L
m,p-Xylene	0.50	1.00	2.00	ug/L
o-Xylene	0.25	0.50	1.00	ug/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Method 8260B)					
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 of DoD QSM 4.1).	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.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 5 of this SOP.	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.
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>2. RSD for RFs for CCCs: VOCs $\leq 30\%$ 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 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 for DoD projects.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value. [$\pm 25\%$ for non-DoD 8260B;]	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 sequence CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. <u>%Difference/Drift for all target compounds and surrogates</u> : VOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ for CCCs only non-DoD 8260B]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8260B, if CCCs exceed, evaluate all analytes for $20\%D$ and qualify as above]	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, holding time has been exceeded or client has approved reporting.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (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 or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	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 qualifier 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 $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected $>RL/LOQ$	Correct problem. 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 client or DoD (appendix G), 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.	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.	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).	Use LCS criteria, above.	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 qualifier 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 LCS acceptance criteria above. MSD or sample duplicate: $RPD \leq 30\%$ or client specified limit (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 qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria			Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	Surrogate	WATER	SOIL	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 qualifier to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
		Dibromofluoromethane	85-120	80-125			
		1,2-Dichloroethane-d4	85-135	75-140			
		Toluene-d8	85-115	80-120			
		Bromofluorobenzene	80-120	80-125			
		QC acceptance criteria specified by DoD (above) or Client. Otherwise, in-house control limits may be used. No limits specified for Method 624.					
Results reported between DL and LOQ	NA.	NA.			NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

Table 5, Tuning Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 6, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8260B/624/8270C/8270D/625 (Circle One)

	Yes	No	NA	Second Review	Level
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1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?

Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).

3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?

4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.

5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?

6. Are the LCS, MS, MSD within control limits and run at the desired frequency?

7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?

8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 7, Internal Standard Association

Analyte	Internal Standard	Analyte	Internal Standard
1,1,1-Trichloroethane	Fluorobenzene	1,1,1,2-Tetrachloroethane	d5-Chlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Fluorobenzene	1,1,2-Trichloroethane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2,3-Trichloropropane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2-Dibromoethane (EDB)	d5-Chlorobenzene
1,1-Dichloropropene	Fluorobenzene	1,3-Dichloropropane	d5-Chlorobenzene
1,2-Dichloroethane	Fluorobenzene	1-Chlorohexane	d5-Chlorobenzene
1,2-Dichloroethane-d4	Fluorobenzene	2-Hexanone	d5-Chlorobenzene
1,2-Dichloroethane (total)	Fluorobenzene	Bromofluorobenzene	d5-Chlorobenzene
1,2-Dichloropropane	Fluorobenzene	Bromoform	d5-Chlorobenzene
1,4-Dioxane	Fluorobenzene	Chlorobenzene	d5-Chlorobenzene
2,2-Dichloropropane	Fluorobenzene	Chlorobenzene-d5	d5-Chlorobenzene
2-Butanone	Fluorobenzene	Dibromochloromethane	d5-Chlorobenzene
2-Chloroethyl vinyl ether	Fluorobenzene	Ethyl Methacrylate	d5-Chlorobenzene
4-Methyl-2-pentanone	Fluorobenzene	Ethylbenzene	d5-Chlorobenzene
Acetaldehyde	Fluorobenzene	m,p-Xylene	d5-Chlorobenzene
Acetone	Fluorobenzene	Methacrylonitrile	d5-Chlorobenzene
Acetonitrile	Fluorobenzene	o-Xylene	d5-Chlorobenzene
Acrolein	Fluorobenzene	Styrene	d5-Chlorobenzene
Acrylonitrile	Fluorobenzene	Tetrachloroethane	d5-Chlorobenzene
Allyl chloride	Fluorobenzene	Toluene	d5-Chlorobenzene
Benzene	Fluorobenzene	Toluene-d8	d5-Chlorobenzene
Bromochloromethane	Fluorobenzene	trans-1,3-Dichloropropene	d5-Chlorobenzene
Bromodichloromethane	Fluorobenzene	Xylenes (total)	d5-Chlorobenzene
Bromomethane	Fluorobenzene	1,1,2,2-Tetrachloroethane	1,4-dichlorobenzene-d4
Carbon disulfide	Fluorobenzene	1,2,3-Trichlorobenzene	1,4-dichlorobenzene-d4
Carbon tetrachloride	Fluorobenzene	1,2,4-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroethane	Fluorobenzene	1,2,4-Trimethylbenzene	1,4-dichlorobenzene-d4
Chloroform	Fluorobenzene	1,2-Dibromo-3-chloropropane	1,4-dichlorobenzene-d4
Chloromethane	Fluorobenzene	1,2-Dichlorobenzene	1,4-dichlorobenzene-d4
Chloroprene	Fluorobenzene	1,3,5-Trimethylbenzene	1,4-dichlorobenzene-d4
cis-1,2-Dichloroethane	Fluorobenzene	1,3-Dichlorobenzene	1,4-dichlorobenzene-d4
cis-1,3-Dichloropropene	Fluorobenzene	1,4-Dichlorobenzene	1,4-dichlorobenzene-d4
Cyclohexane	Fluorobenzene	1,4-Dichlorobenzene-d4	1,4-dichlorobenzene-d4
Dibromofluoromethane	Fluorobenzene	2-Chlorotoluene	1,4-dichlorobenzene-d4
Dibromomethane	Fluorobenzene	4-Chlorotoluene	1,4-dichlorobenzene-d4
Dichlorodifluoromethane	Fluorobenzene	Bromobenzene	1,4-dichlorobenzene-d4
Diisopropyl Ether	Fluorobenzene	cis-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Ethyl tert-Butyl Ether	Fluorobenzene	Hexachlorobutadiene	1,4-dichlorobenzene-d4
Fluorobenzene	Fluorobenzene	Naphthalene	1,4-dichlorobenzene-d4
Hexane	Fluorobenzene	n-Butylbenzene	1,4-dichlorobenzene-d4
Iodomethane	Fluorobenzene	n-Propylbenzene	1,4-dichlorobenzene-d4
Isobutyl alcohol	Fluorobenzene	p-Isopropyltoluene	1,4-dichlorobenzene-d4
Isopropylbenzene	Fluorobenzene	sec-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Acetate	Fluorobenzene	tert-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Methacrylate	Fluorobenzene	trans-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Methyl t-Butyl Ether	Fluorobenzene		
Methylcyclohexane	Fluorobenzene		
Methylene chloride	Fluorobenzene		
Propionitrile	Fluorobenzene		
t-Butyl alcohol	Fluorobenzene		
Tert-Amyl Methyl Ether	Fluorobenzene		
Tetrahydrofuran	Fluorobenzene		
trans-1,2-Dichloroethane	Fluorobenzene		
Trichloroethene	Fluorobenzene		
Trichlorofluoromethane	Fluorobenzene		
Vinyl acetate	Fluorobenzene		
Vinyl chloride	Fluorobenzene		

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 211

REVISION #: 22

EFFECTIVE DATE: 070710

**GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD)
ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB)
BY EPA METHOD 608/608.2 or
SW846 METHOD 8081A/8082 or 8081B/8082A**

APPROVALS:

Lab Director:



Date: 7/8/10

Data Quality Manager:



Date: 7/7/10

Section Supervisor:



Date: 7/7/10

Changes Summary

Revision 22, 07/07/10

- The SOP is an update from Revision 21 dated 04/11/10.
- The SOP has been updated to move specific requirements to tables at the back of the SOP and add Mirex, PCB-1262, PCB-1268 as analytes.

Revision 21, 04/11/10

- The SOP is an update from Revision 20 dated 04/27/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

This SOP is compliant with SW-846 Methods 8000B/8081A/8082 and 8000C/8081B/8082A. *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This Standard Operating Procedure, SOP, is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits

See **Table1**.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Detection Limit/Method Detection Limit, Limit of Detection and Reporting Limit/Limit of Quantitation for each analyte.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition. Analytes are quantitated relative to known standards using the external standard method.

6.0 Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

Section 3.0 of SW-846 Methods 8081A/8082 and Section 4.0 of Methods 8081B/8082A details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308, 309 and 330) to evaluate possible clean-up options for any encountered interferences.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

- 8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
- 8.4 MSDS sheets are available for all reagents and standards that have been purchased. These are located on the bookshelves in the Quality Assurance Officer's office.

9.0 Equipment & Supplies

- 9.1 GC's:
 - 9.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.
- 9.2 Columns:
 - 9.2.1 Restek Siltek Guard Column (or equivalent): 10 meter x 0.32 mm ID
 - 9.2.2 RTX-CLP or ZB-MR1 (or equivalent): 30 meter x 0.32 mm ID x 0.5 µm film thickness fused silica column.
 - 9.2.3 RTX-CLP II or ZB-MR2 (or equivalent): 30 meters x 0.32 mm ID x 0.5 µm film thickness fused silica column.
- 9.3 Autosamplers:
 - 9.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.
- 9.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.
- 9.5 Data Processing Software: Target DB Windows data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the reagents and standards used for the performance of the method. See **Table 5** for information on standard sources/calibration concentrations.
- 10.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the COA and recorded in the LIMS. The date they are opened is recorded in the LIMS along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the LIMS and given a sequential number. The following are noted in the LIMS: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, concentration, expiration date, and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C-4.4°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook.
- 10.3 List of Reagents:
 - Hexane - pesticide quality or equivalent.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples

have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.
- 12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.
- 12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.
- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:
 - 12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
 - 12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
 - 12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.
 - 12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.
- 12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 See Section 14.4 for Calibration details.

14.0 Procedure

- 14.1 The GC/ECD should be primed by injecting a pesticide standard at 200-500 µg/L and/or PCB standard at 2,500 µg/L, 10 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.
- 14.2 Chromatographic conditions:

14.2.1	ZB MR1/MR2 columns:	
	GC	ECD3
	Purge on	60ml/min at 0.50 min.
	Injector/Detector temperature	250/340°C
	Column flow	3.0 mL/min
	Initial column temperature	100°C for 1.0 minutes
	Temperature ramp	35°C/min
	Intermediate column temperature	220°C for 0.0 minutes
	Second Temperature Ramp	15°C/min
	Final Column Temperature	340°C for 2.0 minutes
14.2.2	ZB MR1/MR2 columns:	
	GC	ECD4
	Purge on	60ml/min at 0.50 min.
	Injector/Detector temperature	250/350°C
	Column flow	3.0 mL/min
	Initial column temperature	100°C for 1.0 minutes
	Temperature ramp	35°C/min
	Intermediate column temperature	220°C for 0.0 minutes
	Second Temperature Ramp	15°C/min
	Final Column Temperature	340°C for 2.0 minutes

Note: Current gas chromatograph conditions can be confirmed in the corresponding maintenance log.

14.3 Eval Mix – Before pesticide calibration and/or sample analysis, a degradation check standard (evaluation mix) of endrin and 4,4'-DDT must be injected. Degradation of either compound must not exceed 15 percent. See **Table 2** for criteria and corrective action.

14.4 Calibration - (See SW-846 Method 8000B Section 7.4 or Method 8000C Section 9.3).

14.4.1 Initial Calibration - An initial multi-point calibration curve must be prepared in hexane, analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources and below for makeup of the intermediates. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. For single component pesticides and surrogates, a seven point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Technical Chlordane a single low calibration point standard is analyzed unless they are expected/detected then a six-point calibration is injected and analyzed. Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other Aroclor mixtures (1221, 1232, 1242, 1248, 1254, 1262 & 1268). Full calibration is required if they are expected/detected. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

Mix A/B (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of A/B Mix and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.*

Mirex (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 100µL of Mirex and 50µL Surrogate are injected into a 10mL volumetric flask

containing approximately 9.5mL hexane and diluted to volume with same to make a 1 µg/mL standard.*

Technical Chlordane (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 100µL of Technical Chlordane and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.

Toxaphene (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Toxaphene and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

Aroclor 1016/1260 (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Aroclor 1016/1260 and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

*After capping and inverting several times, all solutions are transferred into labeled, 12ml, teflon-lined, screw-capped vials and stored in the refrigerator at 4°C or less for up to 6 months. These standards are used to make the calibration curve standards in hexane at the concentrations found in table 5.

- 14.4.2 Initial Calibration Verification - A second source standard must be prepared in hexane, analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 14.4.3 Continuing Calibration Verification (CCV) - Every 12 hours (and at the end of the analysis sequence), a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 14.4.4 RT Windows - Retention time criteria set forth in SW-846 method 8000B Section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration using the midpoint standard RTs. If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.
- 14.5 Samples - Prior to using Method 608, SW-846 8081A, 8081B, 8082, 8082A or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3541, 3546, 3640, 3550, 3580, EPA method 608 or CLP).

14.5.1 Example of a sequence run log:

1-Primer A/B Mix-1000 or Primer PCB-10,000
2- EVAL Mix (Pest only)
3- CCV A/B Mix
4- CCV Toxaphene (single point)
5-CCV Chlordane (single point)
6- CCV PCB 1660
7- Method Blank
8-LCS A/B Mix
9-LCS PCB
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample
19-Sample
20-Sample
21-Sample-MS
22-Sample-MSD
23-Sample
24-Sample
25-Sample
26-Sample
27-Sample
28-Sample
29- CCV A/B Mix
30-CCV PCB

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Target DB Windows data system. Quantitative measurements are performed as described in SW-846 8081A Section 7.5.6, and SW-846 8081B Section 11.5.6.1. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Analyte concentration after rounding to 3 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

14.6.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

- 14.6.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your supervisor), [see SOP-308].
- 14.6.4 Analyte quantitation verification.
- 14.7 Identification/Quantitation [See SW-846 method 8081A Section 7.6 or method 8082 Sections 7.7-7.9].
- 14.10.1 Single peak components are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound.
- 14.10.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be qualified appropriately.
- 14.10.1.2 If a compound is outside of its window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to determine if the analyte is present.
- 14.10.2 Multi-peak components (PCB's, Toxaphene and Technical Chlordane) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 7.7.3 of SW-846 Method 8082. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.
- A. Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components.
- B. Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic lab manager's attention.
- C. Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic lab manager's attention.
- 14.10.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. Calculations follow in Section 15.0.

15.0 Data Analysis and Calculations

- 15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.
- 15.2 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

- 15.3 The mean CF is calculated as follows:

$$\text{AvgCF} = \frac{\sum \text{CF for each standard}}{N}$$

- 15.4 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

- 15.5 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration.

- 15.6 Concentration in water samples is calculated as follows:
 [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (μL).

D = Dilution factor, if the sample was diluted prior to analysis.

If no dilution was made, $D = 1$. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (μL). The nominal injection volume for samples and calibration standards must be the same.

CF = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

- 15.7 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

where:

A_x , V_t , D , and CF are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

- 16.1 Method Detection Limit Study or Detection Limit Determination
- 16.2 Limit of Detection Verification
- 16.3 Limit of Quantitation or Reporting Limit Verification
- 16.4 Demonstration of Capability (DOC)
- 16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Please see Waste Disposal, SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

- 21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Method 8081A, 8081B, 8082, 8082A*
- 21.2 *USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 608, 608.2; APX-B*
- 21.3 *USEPA Contract Laboratory Program (CLP) for Organics ILM04.2; ILM04.3*
- 21.4 *DOD Quality Systems Manual, Ver. 3/4.1*

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.
- 22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.
- 22.3 Table 3, Technical Completeness / Accuracy Checklist
- 22.4 Table 4, Data Reviewers Checklist
- 22.5 Table 5, Calibration Standards

Table1- Detection limits

Analyte (water)	MDL/DL	LOD	LOQ/RL	Units
4,4'-DDD	0.00500	0.0100	0.0200	ug/L
4,4'-DDE	0.00500	0.0100	0.0200	ug/L
4,4'-DDT	0.00500	0.0100	0.0200	ug/L
Aldrin	0.00330	0.0100	0.0200	ug/L
alpha-BHC	0.00330	0.0100	0.0200	ug/L
alpha-Chlordane	0.00330	0.0100	0.0200	ug/L
beta-BHC	0.00330	0.0100	0.0200	ug/L
Chlordane (tech)	0.0170	0.0250	0.0500	ug/L
delta-BHC	0.00330	0.0100	0.0200	ug/L
Dieldrin	0.00500	0.0100	0.0200	ug/L
Endosulfan I	0.00330	0.0100	0.0200	ug/L
Endosulfan II	0.00500	0.0100	0.0200	ug/L
Endosulfan sulfate	0.00500	0.0100	0.0200	ug/L
Endrin	0.00500	0.0100	0.0200	ug/L
Endrin aldehyde	0.00500	0.0100	0.0200	ug/L
Endrin ketone	0.00500	0.0100	0.0200	ug/L
gamma-BHC (Lindane)	0.00330	0.0100	0.0200	ug/L
gamma-Chlordane	0.00330	0.0100	0.0200	ug/L
Heptachlor	0.00330	0.0100	0.0200	ug/L
Heptachlor epoxide	0.00330	0.0100	0.0200	ug/L
Methoxychlor	0.00330	0.0100	0.0200	ug/L
Mirex	0.00330	0.0100	0.0200	ug/L
Toxaphene	0.330	0.667	1.00	ug/L
Aroclor-1016	0.125	0.250	0.500	ug/L
Aroclor-1221	0.125	0.250	0.500	ug/L
Aroclor-1232	0.125	0.250	0.500	ug/L
Aroclor-1242	0.125	0.250	0.500	ug/L
Aroclor-1248	0.125	0.250	0.500	ug/L
Aroclor-1254	0.125	0.250	0.500	ug/L
Aroclor-1260	0.125	0.250	0.500	ug/L
Aroclor-1262	0.125	0.250	0.500	ug/L
Aroclor-1268	0.125	0.250	0.500	ug/L
Analyte (Soil)	MDL/DL	LOD	LOQ/RL	Units
4,4'-DDD	0.170	0.340	0.670	ug/Kg
4,4'-DDE	0.170	0.340	0.670	ug/Kg
4,4'-DDT	0.170	0.340	0.670	ug/Kg
Aldrin	0.110	0.340	0.670	ug/Kg
alpha-BHC	0.110	0.340	0.670	ug/Kg
alpha-Chlordane	0.110	0.340	0.670	ug/Kg
beta-BHC	0.110	0.340	0.670	ug/Kg
Chlordane (tech)	0.570	0.850	1.70	ug/Kg
delta-BHC	0.110	0.340	0.670	ug/Kg
Dieldrin	0.170	0.340	0.670	ug/Kg
Endosulfan I	0.110	0.340	0.670	ug/Kg
Endosulfan II	0.170	0.340	0.670	ug/Kg
Endosulfan sulfate	0.170	0.340	0.670	ug/Kg
Endrin	0.170	0.340	0.670	ug/Kg
Endrin aldehyde	0.170	0.340	0.670	ug/Kg

Analyte (Soil)	MDL/DL	LOD	LOQ/RL	Units
Endrin ketone	0.170	0.340	0.670	ug/Kg
gamma-BHC (Lindane)	0.110	0.340	0.670	ug/Kg
gamma-Chlordane	0.110	0.340	0.670	ug/Kg
Heptachlor	0.110	0.340	0.670	ug/Kg
Heptachlor epoxide	0.110	0.340	0.670	ug/Kg
Methoxychlor	0.110	0.340	0.670	ug/Kg
Toxaphene	11.0	22.0	33.0	ug/Kg
Aroclor-1016	4.17	8.33	16.7	ug/Kg
Aroclor-1221	4.17	8.33	16.7	ug/Kg
Aroclor-1232	4.17	8.33	16.7	ug/Kg
Aroclor-1242	4.17	8.33	16.7	ug/Kg
Aroclor-1248	4.17	8.33	16.7	ug/Kg
Aroclor-1254	4.17	8.33	16.7	ug/Kg
Aroclor-1260	4.17	8.33	16.7	ug/Kg
Aroclor-1262	4.17	8.33	16.7	ug/Kg
Aroclor-1268	4.17	8.33	16.7	ug/Kg
Analyte (TCLP)	MDL/DL	LOD	LOQ/RL	Units
Chlordane (tech)	0.000170	0.000250	0.000500	mg/L
Endrin	0.0000500	0.000100	0.000200	mg/L
gamma-BHC (Lindane)	0.0000330	0.000100	0.000200	mg/L
Heptachlor	0.0000330	0.000100	0.000200	mg/L
Heptachlor epoxide	0.0000330	0.000100	0.000200	mg/L
Methoxychlor	0.0000330	0.000100	0.000200	mg/L
Toxaphene	0.00330	0.00670	0.0100	mg/L

Table 1. Organic Analysis by Gas Chromatography (Methods 8081, 8082)

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.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
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. Minimum ± 0.030 min.	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.
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 for DoD analyses. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration, if detected. Results may not be quantitated using a single point.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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. All project analytes within $\pm 20\%$ of expected value from the ICAL;	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate for DoD analyses.	Problem must be corrected. No samples may be run until calibration has been verified.
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. All project analytes within $\pm 20\%$ 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 qualifier 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.	Correct problem, then see SOP QS05. 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.	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 qualifier 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.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	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 qualifier 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.	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 qualifier 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.	Qualify surrogate results on form I.	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.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA.	Apply qualifier 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.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8081/8082

	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
3. Is the low standard equal to or below the MRL/LOQ?				
4. Are the %RSD or fit criteria within QC limits for all analytes?				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				
6. Are surrogate recoveries within QC limits? (one surrogate both columns)				

ANALYST DATA REVIEW CHECKLIST, cont.

E. QC Samples

- 1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs? _____
- 2. Is the Laboratory Control Sample and its percent recovery within QC limits? _____
- 3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits? _____

F. Others

- 1. Are all nonconformances included and noted? _____
- 2. Are all calculations checked at the minimum frequency with one example worked out in the space below? _____
- 3. Did analyst initial/date the appropriate printouts and report sheets? _____
- 4. Are all sample IDs and units checked for transcription errors? _____
- 5. Are all manual integrations checked by a second reviewer to verify they were performed correctly? _____

Calculation – one complete calculation from raw area/height to final concentration:

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 – Standard concentrations/sources
NOTE: All standards are fully documented within the LIMS

	Level 1 (ppb)	Level 2 (ppb)	Level 3 (ppb)	Level 4 (ppb)	Level 5 (ppb)	Level 6 (ppb) MIDPOINT	Level 7 (ppb)	Primary Source (Concentration-ppm)	Secondary Source** (Concentration-ppm)
Single Component Pesticides	1	5	10	25	50	100	200	Restek (200)	Accustandard (1000)
Mirex	1	5	10	25	50	100	200	Accustandard (100)	ChemService (100)
DCB/TCMX	1	5	10	25	50	100	200	Restek (200)	NA
Technical Chlordane*	-	5	10	25	50	100	200	Restek (1000)	Ultra Scientific (5000)
Toxaphene*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (100)
PCB-1016/PCB-1260	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1221*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1242*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1248*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1254*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1262*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (100)
PCB-1268*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (500)

* - Toxaphene and Technical Chlordane single point at low standard unless detected. PCB calibration 1016/1260 unless other pattern detected.

** - Secondary Source may be from any vendor other than the primary source.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 218

REVISION #: 07

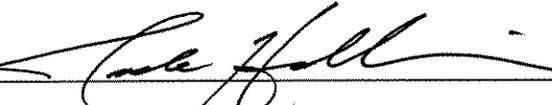
EFFECTIVE DATE: 20100907

1, 2-DIBROMOETHANE AND 1, 2-DIBROMO-3-CHLOROPROPANE BY
GC/ECD EPA METHODS 504.1 & SW-846 8011

APPROVALS:

Lab Director:  Date: 9/8/10

Data Quality Manager:  Date: 9/8/10

Section Supervisor:  Date: 9/8/10

Changes Summary

Revision 07, 08/24/10

- The SOP is an update from Revision 06 dated 09/30/09
- References to acceptance criteria were moved to table 2 and references to table 2 were added in the body of the SOP.
- F-tables from DoD have been added and updated to include other method references.
- Analyst checklist has been added.

Revision 06, 09/30/09

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with EPA method 504.1 and SW-846 method 8011.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to the determination of 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in drinking water and groundwater.

3.0 Detection Limit

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,2-Dibromo-3-chloropropane (DBCP)	0.0100	0.0200	0.0300	ug/L
1,2-Dibromoethane (EDB)	0.0100	0.0200	0.0300	ug/L

4.0 Scope of Application, Including Components to Be Analyzed

- 4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed above.
- 4.2 The GC/ECD system is used to analyze 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in ground water. This SOP will describe the analysis of these compounds using a temperature programmable gas chromatograph configured with dual electron capture detectors (ECD) and a capillary column splitless injector.
- 4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 Thirty-five mL of sample are extracted with 2 mL of hexane. One μ L of the extract is then injected into the gas chromatograph equipped with dual linearized electron capture detectors for separation and analysis. All standards, QC spikes, and blanks are prepared, extracted, and analyzed using this same method.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

- 7.1 Interferences can come from the extracting solvent or reagents. Reagent Blanks must be analyzed to check for interferences with the analysis.
- 7.2 Interferences can come from other organic compounds contained in the sample.
- 7.3 Dibromochloromethane is a common chlorinated drinking water contaminant, which in high concentrations may mask low concentrations of EDB. Therefore, special care should be taken in the identification and confirmation of EDB.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

9.0 Equipment & Supplies

- 9.1 Syringes – 10ul, 25ul, 100ul, and 5ml (Hamilton 702N or equivalent)
- 9.2 Vials – 2ml amber autosampler with Teflon-lined screw cap; 40ml VOA with Teflon-lined screw cap; 12ml with Teflon-lined screw cap

- 9.3 Transfer Pipets
- 9.4 Graduated Cylinder (Glass) – 50ml
- 9.5 Volumetric Flask – 10ml
- 9.6 Balance – top loading, 0.01g
- 9.7 Gas Chromatograph – Agilent 6890 (temperature programmable)
- 9.8 Autosampler – HP-7683 injector
- 9.9 Columns
 - 9.9.1 Column A – Phenomenex ZB-Multiresidue-1, 30m, 0.32mm ID, 0.50um thickness or equivalent.
 - 9.9.2 Column B (confirmation column) – Phenomenex ZB-Multiresidue-2, 30m, 0.32mm ID, 0.25um thickness or equivalent.
- 9.10 Data System
 - 9.10.1 Acquisition Software – HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.
 - 9.10.2 Data Processing Software – TARGET Chemserver 4920 data system is interfaced to the HP Chemstation. The system accepts, processes, and stores acquired data.

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

- 10.2 Organic-free reagent water from our laboratory modulab analytical purification system.
- 10.3 Hexane - Pesticide grade
- 10.4 Methanol – Purge and Trap.
- 10.5 Sodium Chloride - demonstrated to be interference free. If needed, sodium chloride can be placed in the muffle furnace at room temperature and increase to 400° for 30min. Place in a bottle and cap.
- 10.6 Intermediate stock standards are prepared from these certified stock standards. All intermediate standards (in methanol) are prepared at a concentration that can be easily diluted (see below) to prepare aqueous calibration standards that will bracket the working calibration range. The information concerning the preparation of these standards is noted in the LIMS system, detailing how they were made, solvent (methanol, laboratory reagent blank water) used, date made, expiration date and given a unique sequential number Standards may be used for at least four weeks. A second source check is required every time a new standard or standard curve is implemented. The intermediate stock standard is then stored at 4 C.
- 10.7 Calibration standards must be prepared at a minimum of five concentration levels for each analyte through dilution of the intermediate stock standard into laboratory reagent blank

water. One of the concentration levels should be near, but above, the method detection limit (MDL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or define the working range of the GC-ECD system. Typical concentration levels are 0.03, 0.05, 0.10, 0.15, and 0.20 µg/L. In order to prepare accurate aqueous standard solutions, the following precautions must be observed:

- 10.7.1 Use a 25 µL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).
- 10.7.2 Rapidly inject the alcoholic standard into the filled volumetric flask or vial. Remove the needle as fast as possible after injection.
- 10.7.3 Mix the aqueous standards by inverting the flask or vial three times only.

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Samples are collected in 40ml vials and stored at 4°C.
- 11.3 Holding time is 14 days from sample collection to extraction and analysis.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.2 An initial demonstration must be performed by each analyst performing this method. Four LCSs are analyzed at 0.50ug/L. See **Table 2** for acceptance criteria.
- 12.3 For method 504.1, each day a low level standard at 0.02ug/L should be analyzed to check for adequate sensitivity.
- 12.4 A CCV (QC check standard) at a concentration of 0.50 µg /L with both analytes is required each batch or at a 5% frequency whichever comes first. **See table 2** for acceptance criteria and corrective action.
- 12.5 An LCS (QC reference sample) is required each batch at a concentration of 0.50 µg/L to check the calibration standard. **See table 2** for acceptance criteria and corrective action.
- 12.6 Check the performance of the analytical system daily by using data gathered from analyses of blanks, standards, and replicate samples. System maintenance can be indicated through the performance of the analyses.
 - 12.6.1 Peak tailing problems which show up in the chromatography are traceable to active sites on the GC column or to the detector.
 - 12.6.2 Check precision between replicate analyses. The system should perform with an average relative standard deviation of less than 10%.

Poor precision could indicate pneumatic leaks, especially at the injection port.

- 12.6.3 Maintenance of the GC system should be performed and logged into the maintenance logbook. Maintenance should include the following:
 - 12.6.3.1 Clean or deactivate glass injection port insert or replace with a cleaned and deactivated insert.
 - 12.6.3.2 Break off the first few inches of the injection port side of the column.
 - 12.6.3.3 Remove the column and column back-flush according to the manufacture's specifications.
 - 12.6.3.4 If all else fails to correct the problem, the metal injection port body may need to be deactivated or the column replaced.

- 12.7 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program). If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.
- 12.8 Sample analyses will begin after the analyst performs the various checks just mentioned. If there was a problem in meeting the QC criteria then system maintenance may be required (see above) as stated.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 An extracted five-point calibration curve must be introduced and analyzed for each analyte using the appropriate instrument parameters. One level of concentration containing EDB and DBCP should be near but above the MDL for each compound (reference SW-846 Method 8011 Section 7.2).
 - 13.2.1 Tabulate peak height or area response versus the concentration in the standard and prepare a calibration curve for each compound. **See table 2** for acceptance criteria and corrective action. All calibration integrations must be verified and any manual integrations are documented by the inclusion of the chromatogram (which includes the before and after chromatograms of the peak integrations) with the quantitation report. See **SOP QS07** for guidance.
 - 13.2.2 An Initial Calibration Verification (ICV) standard is prepared in the same manner as the QC reference sample and analyzed after the initial calibration but before client samples. The ICV must pass acceptance criteria (see **Table 2**) before the analysis of samples can continue.

14.0 Procedure

- 14.1 Samples and standards are removed from storage and left to equilibrate to room temperature. Sample and/or standard extraction must be performed in an organic solvent free environment.
- 14.2 For samples and field blanks contained in a 40 mL VOA vial remove the vial cap and measure off and discard 5 mL of sample using a 5 mL transfer pipette. Replace the vial cap and weigh and record the weight of the container with contents to the nearest 0.1 g. This weight will be used for subsequent sample volume determination (see below).
- 14.3 For calibration standards, check standards, QC reference samples, and blanks, measure a 35 mL volume (laboratory reagent blank containing the appropriate spike) using a 50 mL graduated glass cylinder and transfer to a 40 mL vial.
- 14.4 Begin the extraction of the samples by removing the cap again and adding 7 g of NaCl to all samples. Recap the sample container and dissolve the NaCl by shaking by hand for about 20 seconds
- 14.5 Remove the cap again and using a 1mL syringe, add 2.0 mL of hexane. Recap and shake vigorously by hand for 1.0 minute. Allow the water and hexane to separate (if stored at this stage, keep the container upside down).
- 14.6 Remove the cap and carefully transfer a sufficient amount (0.5 - 1.0 mL) of the hexane layer into a vial using a disposable glass pipette. Repeat this step to a second vial being careful

not to include any of the water phase. Reserve this second vial at 4 ° C for reanalysis if necessary.

- 14.7 Determination of the sample and field blank volume is done by totally removing any sample/hexane mixture left in the vial (this is discarded). Re-weigh the empty vial including the cap and calculate the net weight of sample by the difference to the nearest 0.1 g from the previous weight determination.

15.0 Data Analysis and Calculations

- 15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.
- 15.2 GC oven conditions
 - 15.2.1 Injector temperature: 200°C.
 - 15.2.2 Detector temperature: 290°C.
 - 15.2.3 Carrier gas (Helium) 1.9 ml/min. constant flow
 - 15.2.4 Temperature program:
Initial temperature: 35°C, hold for 2.0 min.
Program: Ramp at 12°C / min. to 290°C
- 15.3 Transfer all samples (including standards, blanks, and unknown samples) to an autosampler set up for a 1.0 µL injection. Analyze according to previously stated instrument parameters (see above).
- 15.4 Analytes are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. If both columns are equivalent, the higher concentration is reported.
- 15.5 Samples are reduced using the TARGET Chemserver data system. The analytes detected in the samples are calculated using the calibration factor or calibration curve giving an uncorrected concentration of each analyte in the sample (reference SW-846, Method 8011, Section 7.7). These results are then referenced to any dilutions and the initial sample volume to complete the quantitation of analyte in the sample. The final analytical results are reported in µg / L.
- 15.6 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, and/or Technical Director or Quality Assurance Manager.

16.0 Method Performance

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See [Table 2](#) for acceptance criteria.

See method 8011 and 504.1 for method performance.

17.0 Pollution Prevention

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8011.*

21.2 *Method 504.1, 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-Trichloropropane in Water by Microextraction and Gas Chromatography, USEPA, 1995.*

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, included in section 3.0.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

Table 1. Organic Analysis by Gas Chromatography (Method 504.1/8011)

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.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
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 or 0.03min., whichever is greater.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes (504.1 only requires 3 points)	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$ ($<10\%$ for non-DoD 8011 projects) 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 for DoD projects.
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.	

Table 2. Organic Analysis by Gas Chromatography (Method 504.1/8011) Cont'd

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects), and at the end of the analysis sequence.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL;	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should 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 $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, 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 client or DoD (appendix G), 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.	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.

Table 2. Organic Analysis by Gas Chromatography (Method 504.1/8011) Cont'd

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria above.	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 qualifier 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 LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply P-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.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 504.1/8011

	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of five Calibration Standards?	_____	_____	_____	_____
2. Is the low standard at or below the LOQ/RL?	_____	_____	_____	_____
3. Are the % RSDs within QC limits for all analytes?	_____	_____	_____	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?	_____	_____	_____	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples (10 for DoD) and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
D. Sample Analysis				
1. Are all sample holding times met?	_____	_____	_____	_____
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	_____	_____	_____	_____
3. Are all compounds identified on the primary column confirmed on the secondary column?	_____	_____	_____	_____
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than 1/2 the LOQ/RL?	_____	_____	_____	_____
2. Is the LCS extracted at the desired frequency and are the percent recoveries within QC limits?	_____	_____	_____	_____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries/RPDs within QC limits?	_____	_____	_____	_____
F. Others				
1. Are all nonconformances included and noted?	_____	_____	_____	_____
2. Are all calculations checked at the minimum frequency with an example calculation included each batch?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Do all manual integrations have before/after intialed/dated/coded and checked by a second reviewer to verify why they were performed?	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 225

REVISION #: 09

EFFECTIVE DATE: 20100907

**GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING
SW-846 METHOD 5035/5035A FOR 8260B ANALYSIS**

APPROVALS:

Lab Director:  Date: 9/8/10

Data Quality Manager:  Date: 9/8/10

Section Supervisor:  Date: 9/8/10

Changes Summary

Revision 09, 09/07/10

- The SOP is an update from Revision 08 dated 09/24/08
- The SOP has been updated to include reference to 5035A and preservation by freezing for unpreserved Terracores and Encores.

**GC/MS - VOLATILE
NON - AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 5035/A**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035 and 5035A. Soil samples should be sampled in the field using the EnCore™ sampler or prepared VOA vials (sometimes referred to as Terracore samplers) then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

2.0 SUMMARY

Samples are collected in EnCores or prepared VOA vials and submitted to the laboratory for preparation/analysis.. EnCore samplers have to be frozen or prepared within 48 hrs of collection. Prepared VOA vials (sometimes referred to as Terracores) are shipped already prepared in water, methanol or preservative solution. If prepared in water, freezing is required within 48 hours. If preservative is used, refrigeration is the only requirement.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

EnCores are prepped within 48 hrs of collection or frozen until preparation can be completed. Preparation can be in sodium bisulfate with refrigeration at 4°C or in reagent water with freezing. Prepared VOA vials are received already prepared in water, methanol or sodium bisulfate solution. If prepared in water, freezing is required within 48 hours of collection. If preservative is used, refrigeration at 4°C is the only requirement. Holding Time is 14 days from collection once preserved.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

5.0 EQUIPMENT AND MATERIALS

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH₃OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, NaHSO₄ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of NaHSO₄ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the NaHSO₄ in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

7.1 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

7.2 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the “low-level” sample. (Some projects may not require the “low-level” detection limits, in this case only the high level sample preparation would be required.). A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. **Additional bulk samples should be collected for screening and dry weight determination without the preservative.** Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

8.0 PROCEDURE

- 8.1** Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.
- 8.2** The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.
- 8.3** Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:
 - 8.3.1** Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.
 - 8.3.2** Get the samples from the Hobart assigned for volatile sample storage and log them out.
 - 8.3.3** Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).
 - 8.3.4** Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial

to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ± 0.01 g of this value before using for sample preparation.

- 8.3.5** For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate an NCR to document the problem.

- 8.3.6** Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.
- 8.3.7** Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

9.0 ANALYSIS

- 9.1** Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.
- 9.2** Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

9.2 If an extra bulk jar was not received for percent moisture determination, an alternate procedure using the methanol vial can be used with advance notice:

- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids (recommend a tare weight on the vial to the same degree of accuracy).
- Preserve the vial as normal.
- After we know the methanol extract is not needed or has been analyzed successfully, allow the methanol to evaporate and dry as necessary for % solids determination.
- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids.

10. HEALTH, SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the Quality Assurance Officers office.

10.4 Please see Waste Disposal SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

REFERENCES

1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035, December 1996.

1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Other Methods; Method 5035A, July 2002.

DEFINITIONS

Refer to SOP QS08 for common environmental laboratory definitions.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 300

REVISION #: 18

EFFECTIVE DATE: 042610

**GC/MS SEMI-VOLATILE
BNA-AQUEOUS MATRIX
EXTRACTION USING
SW-846 METHOD 3510C
FOR 8270/625 ANALYSIS**

APPROVALS:

Lab Director:  Date: 4/27/10

Data Quality Manager:  Date: 4/27/10

Section Supervisor:  Date: 4/27/10

Changes Summary

Revision Date: 042610

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 625.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples.

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated to an appropriate final volume.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 “Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.

7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.

7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

9.1 Separatory Funnel – 2L with Teflon stopcock

9.2 Beaker – 250mL or 400mL

9.3 Drying/Chromatographic column – 20mm I.D. x 300mm

9.4 Filter funnel

9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent

9.6 Metal rack – capable of holding six glass evaporation tubes

- 9.7 Turbo-Vap Evaporator – heated and capable of temperature control (+5°C); the bath should be vented into a hood
- 9.8 Vials, 2.0 mL glass with Teflon-lined screw cap
- 9.9 pH indicator paper – wide range (1.0-12.0)
- 9.10 Syringe – 1mL
- 9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
- 9.12 Pasteur pipette – length 9”
- 9.13 Pasteur pipette bulb
- 9.14 Labels – DYMO
- 9.15 Teflon Bottles – 500mL
- 9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
- 9.17 Ring Stand – 3-prong
- 9.18 Burette clamp – double
- 9.19 Aluminum foil – heavy duty
- 9.20 Nitrogen tank – equipped with pressure regulator
- 9.21 Boiling chips – Teflon
- 9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

- 10.1 Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab as needed.
- 10.2 Sodium Hydroxide Solution - (10N). Weigh 800g NaOH (purchased in a fiber drum from Tennessee Reagents # 2-31825-25lb or equivalent) into a 2000mL volumetric flask and add approximately 1000mL of reagent water. Swirl until pellets are mostly dissolved. Add a stir bar and place on stir plate. This mixture will get very hot. Continue to add reagent water while mixture is being stirred until a final volume of 2000mL is attained. Let stand until cool. Transfer to 1000mL Teflon containers.
- 10.3 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh (purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent). For low level tests, place an aliquot in a 1500mL heavy duty Pyrex beaker and bake in muffle furnace at 400°C for a minimum of 4 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature
- 10.4 Glass Wool – Roving , 9989 Glass (purchased from Fisher #11-388 or equivalent).
- 10.5 Sulfuric Acid Solution - (1:1), slowly add 500mL of H₂SO₄ (Fisher, suitable for trace metal analysis #A300C-212 or equivalent) to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Allow to cool before use.
- 10.6 Extraction Solvent - Methylene Chloride (purchased from Fisher #D151-4 or equivalent) Please read SOP-336 before handling this solvent in our laboratory.
- 10.7 The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.
 - 10.7.1 BNA Surrogate** – The base neutral and acid surrogate are mixed together in one solution (purchased from NSI #WL-371-C at concentrations of 100-200ug/mL). The expiration for this standard is 6 months from the date opened. Use 0.5mL of this solution per 1000mL of aqueous sample.

- 10.7.2 BNA Spiking Solution** – The base neutral and acid spiking solutions are mixed together in one solution called BNA LCS#1 (This spiking solution contains all the compounds that are normally calibrated by GC/MS). This solution, with a final concentration of 100ug/mL, is prepared in Methanol by making a dilution of stock purchased from reputable vendors (BNA LCS #1 spike kit #K-943 and 1-methylnaphthalene #1288-01-08 are purchased from NSI, 2,6 Dichlorophenol #95591 is purchased from Absolute Standards and 1,4 Dioxane #30287 is purchased from Restek). Use 0.5mL of this solution per 1000mL of aqueous sample. Another spiking solution is also used, called BNA LCS#2. This solution contains short or matrix spike list base extractable compounds. This solution, with a final concentration of 100ug/mL, is prepared in Methanol by making a dilution of stock purchased from NSI #Q-6104-0. Use 0.5mL of this solution in combination with BNA LCS#1 for all full list BNA requirements. BNA LCS #2 may be omitted from samples requiring PAH analysis. (For low level PAHs, use 1.0mL of a 1.0ug/mL solution made from BNA LCS #1, called “LLPAH spiking solution.”) All standards expire 6 months from the date they are made.
- 10.7.3 BNA TCLP Spike** – 0.5mL of BNA LCS#1 and BNA LCS#2 is added per 100mL volume. This volume is provided by Wet Chemistry in a 1L glass amber bottle. 100mL is removed from this container and measured using a graduated cylinder.

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not Applicable to this SOP

14.0 Procedure

- 14.1 All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
- 14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS.
- 14.1.2 This backlog is used to determine extraction priorities based on hold times and due dates.
- 14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.

- 14.1.4 Samples are placed in LIMS “batches” based on parameter and extracted accordingly.
- 14.2 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:
- 14.2.1 SLUDGE** - use only 100mL and dilute to 1000mL with reagent water.
- 14.2.2 TCLP EXTRACT** - use only 100mL and dilute to 1000mL with reagent water. A separate matrix spike of 100mLs should be set up at the same time. Dilute to 1000mL with reagent water.
- 14.2.3 1BAD MATRIX** – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. SPLP extract-use 1 liter.
- 14.2.4 NPDES client** - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1mL is added to the LCS and the matrix spike.
- 14.2.5 ACID EXTRACT WITH BAD MATRIX** - a cleanup step is added. Samples are taken to a high pH, extracted with 60mL methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.
- 14.3 LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs)** – Samples require a BNA extraction. Use the surrogate and spiking solution specified.
- 14.4 Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the LIMS bench sheet and, later, in LIMS.
- 14.5 Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100mL and dilute to 1000mL with reagent water. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed. Rinse separatory funnels with methanol. Place label from sample bottle onto separatory funnel as samples are poured into funnels to ensure proper identification. Use Avery labels to properly identify method blank, LCS, and LCSD.
- 14.6 Using the 1000mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.

- 14.7 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.
- 14.8 Generally 0.5mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. Someone must verify that the surrogate has been added by initialing LIMS bench sheet.
- 14.8.1 NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.
- 14.9 For the sample in each analytical batch selected for spiking, use the 0.5mL glass syringe designated for BNA spike, to add 0.5mL of BNA spiking solution. **For low level PAHs use 1.0mL of the 1.0ppm LLPAHs spiking solution.** Someone must verify that the spike has been added by initialing the LIMS bench sheet. For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.
- 14.10 Enter the ID# of the surrogate/spike used on the LIMS bench sheet and, later, in LIMS.
- 14.11 ACID EXTRACTION: Adjust the pH to between 1.0 and 2.0, using 2mL of 1:1 H₂SO₄. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H₂SO₄ solution in small increments, as required to attain the proper pH.
- 14.12 Add 40mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.
- 14.13 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.
- 14.13.1 NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.
- 14.14 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40mL into a 250mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 250mL glass beaker.
- 14.15 Following Steps 14.12 through 14.14, extract two more times with 40mL of methylene chloride. Combine the three solvent extracts into the same 250mL beaker.
- 14.16 BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater, using 10N NaOH. Start by adding 5.0mL to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in

small increments, as required to attain the proper pH. BNA extraction is necessary when doing low level PAHs.

14.16.1 NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.

14.17 FOR 8270 extraction: Extract one more time with 40mL of methylene chloride following Step 14.16. Do not combine BN and Acid extracts in a same 250mL beaker. However, you may filter BN and Acid extracts through the same sodium sulfate filter and combine into the same turbo in order to concentrate BN and acid extracts for one final extract.

14.17.1 NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.

14.18 For 625 extractions: extract 3 more times with 40 mL methylene chloride following steps 14.12 through 14.14. Combine BN extracts in the empty 250mL sample beaker as the acid portion concentrates in the turbo vap. Following step 14.24, concentrate the acid extract to ~5mL and then filter the BN extract into the same turbo.

14.19 Prepare to dry the sample by either of the following methods:

14.19.1 Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR

14.19.2 Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

14.20 If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

- 14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of Na₂SO₄ by sprinkling the top layer with Na₂SO₄ until it hardens, separates, and drops to the bottom.
- 14.22 Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume on the LIMS bench sheet to the nearest 10-mL and record, later, in LIMS.
- 14.23 Prepare sample vial tray using labels printed off from LIMS that identify sample numbers, initial/final volumes, client, parameter, and date extracted.
- 14.24 **TURBO-VAP CONCENTRATION**
- 14.24.1 Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.
- 14.24.2 Record the numbers of the Turbo-Vap tube on the LIMS bench sheet and place the tube in a metal holder.
- 14.24.3 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.
- 14.24.4 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- 14.24.5 When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. **THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!!** A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- 14.24.6 Draw ~0.25 mL of methylene chloride into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 2-mL vial. Add methylene chloride from the designated clean pipette and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

- 14.25 The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.
- 14.26 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations
Not Applicable to this SOP

16.0 Method Performance

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures
Not Applicable to this SOP

19.0 Contingencies for Handling out of control or unacceptable data
Not Applicable to this SOP

20.0 Waste Management

- 20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

- 21.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition
21.2 40 CFR, Method 625.

22.0 Tables, Diagrams, Flowcharts, and Validation Data
Not Applicable to this SOP

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 302

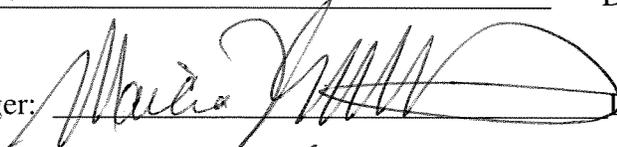
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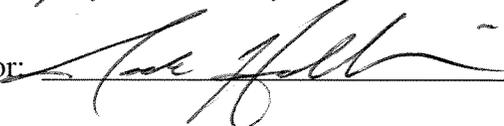
EFFECTIVE DATE: 042610

**PESTICIDE/PCBs
AQUEOUS MATRIX EXTRACTION
FOR EPA METHOD 608/608.2 AND
SW846 METHOD 8081/8082
USING SW846 METHOD 3510C**

APPROVALS:

Lab Director:  Date: 4/27/10

Data Quality Manager:  Date: 4/27/10

Section Supervisor:  Date: 4/27/10

Changes Summary

Revision Date: 042610

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 608/608.2

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated and exchanged to hexane.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

6.2.1 PCBs- polychlorinated biphenyls

6.2.2 Pest- pesticides

6.2.3 TCMX- tetrachloro-m-xylene

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.

7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.

7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

- 9.1 Separatory Funnel – 2L with Teflon stopcock
- 9.2 Beaker – 250mL or 400mL
- 9.3 Drying/Chromatographic column – 20mm I.D. x 300mm
- 9.4 Filter funnel
- 9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent
- 9.6 Metal rack – capable of holding six glass evaporation tubes
- 9.7 Turbo-Vap Evaporator – heated and capable of temperature control ($\pm 5^{\circ}\text{C}$); the bath should be vented into a hood
- 9.8 Vials, 10mL glass with Teflon-lined screw cap
- 9.9 pH indicator paper – wide range (1.0-12.0)
- 9.10 Syringe – 1mL
- 9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
- 9.12 Pasteur pipette – length 9”
- 9.13 Pasteur pipette bulb
- 9.14 Labels – Avery
- 9.15 Teflon Bottles – 500mL
- 9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
- 9.17 Ring Stand – 3-prong
- 9.18 Burette clamp – double
- 9.19 Aluminum foil – heavy duty
- 9.20 Nitrogen tank – equipped with pressure regulator
- 9.21 Boiling chips – Teflon
- 9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

- 10.1 Reagents
 - 10.1.1 Reagent water – Reagent water is gathered in a carboy from source in the instrument lab daily.
 - 10.1.2 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent. Place an aliquot in a 1500mL heavy-duty Pyrex beaker and bake in muffle furnace at 400°C for a minimum of 4 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature.
 - 10.1.3 Sulfuric Acid Solution (1:1) – Slowly add 500mL concentrated Sulfuric Acid, purchased from Fisher #A300C-212 or equivalent, to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Let stand until cool.
 - 10.1.4 Sodium Hydroxide Solution (10N) – Weigh 800g NaOH, purchased in a fiber drum from Tennessee Reagents #2-31825-25lb or equivalent, into a 2000mL volumetric flask and add approximately 1000mL of reagent water. Swirl until pellets are mostly dissolved. Add a stir bar and place on stir plate. This mixture will get very hot. Continue to add reagent water while mixture is being stirred until a final volume of 2000mL is attained. Let stand until cool. Transfer to 1000mL Teflon containers.

- 10.1.5 Methylene Chloride - purchased from Fisher #D151-4 or equivalent. **Please see SOP 336 before handling this solvent in our laboratory.**
- 10.1.6 Hexane – suitable for gas chromatography, purchased from Fisher #H303-4
- 10.2 Standards – The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.
- 10.2.1 TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) – Surrogate solution is prepared, with a final concentration of 0.5ug/mL, by diluting a stock solution (purchased from Restek #32000) in acetone. This solution is named “Pesticide Surrogate for Extractions 500ppb” and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample.
- 10.2.2 PCB Spiking Solution – For all standard extractions, a mixture of 1016/1260 is prepared and used. The stock standards (purchased by Accustandard 1016 #APP-9-158-10X and 1260 #C260S-H-10X) are diluted in acetone to a final concentration of 5ug/mL. This solution is named “PCB 1660 LCS for Extractions 5ppm” and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. The Laboratory Director and/or Organic Manager will determine if another PCB mixture is necessary, such as 1242, 1258, or 1254.
- 10.2.3 Pesticide Spiking Solution – A spiking solution, with a final concentration of 1ug/mL, is prepared by making a dilution of the Pesticide AB ICV Intermediate (this is made in-house by GC operators) in acetone. This solution is named “Pesticide AB LCS for Extractions 1.0ppm” and expires 2 weeks after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. For 608 samples, 1 out of every 10 samples must be spiked
- 10.2.4 TCLP- When necessary to set up a TCLP, in addition to setting up the sample, two matrix spikes must be set up and should include the following:
- A. TCLP Spike 1 – This matrix spike must include a solution containing Chlordane at a concentration of 100ug/mL and Toxaphene at a concentration 10ug/mL. Both compounds are diluted in acetone from stock standards purchased from reputable vendors (Chlordane from Ultra Scientific #EPA-1086, Toxaphene from AccuStandard #P-0935-H). This solution is named “Tox/Chlor LCS for Extractions 10-100ppm” and expires 6 months from the date it is made. Add 1.0mL of leachate.
 - B. TCLP Spike 2 – This matrix spike must include the Pesticide Spiking Solution known as “Pesticide AB LCS for Extractions 10ppm.” Add 1.0mL of this solution per 100mL of leachate.

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not applicable to this SOP

14.0 Procedure

- 14.1 All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary to extract from the following (Note: never extract samples of unknown origin without discussion with supervisor):
 - 14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS
 - 14.1.2 This backlog is used to determine extraction priorities based on hold times and due dates.
 - 14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.
 - 14.1.4 Samples are placed in LIMS “batches” based on parameter and extracted accordingly.
- 14.2 Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid.
- 14.3 Find out if any special dilutions need to be made for client. Routine procedures for difficult matrices are listed below:
 - 14.3.1 Sludge – use only 100mL and dilute to 1000mL with reagent water
 - 14.3.2 TCLP Extract – use only 100mL for the sample and dilute to 1000mL with reagent water. There must be two matrix spikes of 100mL as well that are also diluted to 1000mL with reagent water.
 - 14.3.3 Bad Matrix – e.g. a liquid that is partially sediment. See Organics Supervisor to find out what dilution, if any, should be made.
 - 14.3.4 NPDES client – Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0-9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9 using NaOH solution or Sulfuric Acid, as necessary) or to extract the sample within 72 hours of sampling. If a pH adjustment is made, the details of the adjustment must be recorded on the sample COC and in LIMS. Set up one full list matrix spike for every ten samples.
- 14.4 Mark the amber glass container of each sample at the water meniscus with “white out” for later determination of sample volume.

- 14.5 Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the bench sheet and later, in LIMS.
- 14.6 Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and a LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Follow instructions for TCLP in section 14.3.2 of this SOP. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed.
- 14.7 Rinse separatory funnels with methanol and discard of waste according to SOP QS14.
- 14.8 Pour samples into separatory funnel, placing the label from the sample bottle on the designated separatory to ensure proper identification. Use Avery labels to properly identify method blank, LCS, LCSD, any TCLPs, and TCLP spikes. If a sample requires both Pesticide and PCB analysis, a Pesticide LCS/MS/MSD (if client specified) or LCS/LCSD and a PCS LCS/MS/MSD (if client specified) or LCS/LCSD must be processed to satisfy QC requirements for the batch.
 - 14.8.1 Due to limited volume received, it is usually necessary to use 500mL of sample to do a matrix spike so that a matrix spike duplicate can also be extracted. If only one sample container is provided for spiking purposes, use a 500mL glass cylinder to measure out half of the sample for extraction. Add half of the normal amount of spiking solution and half of the normal amount of surrogate.
- 14.9 Add 50mL of methylene chloride to the empty sample container, swirl, and pour into the designated separatory funnel.
- 14.10 Using the 1L glass graduated cylinder marked "DIH20 WATER ONLY" measure 1L of reagent water from the carboy and transfer it to the designated separatory funnels for method blank, LCS, and LCSD.
- 14.11 Add 50mL of methylene chloride to the method blank, LCS, and LCSD.
- 14.12 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set the surrogate/spike out at least ten minutes before use to allow it to warm to room temperature.
- 14.13 Using the 1.0mL glass syringe marked "TCMX/DCB" surrogate, add 1.0mL of TCMX/DCB surrogate to each sample, method blank, and spike. A second analyst must verify that the surrogate has been added. Enter the ID# of the standard, amount, and the initials of the analysts on the LIMS generated bench sheet and later in LIMS.
- 14.14 Determine if the sample will require a Pesticide spike, PCB spike, or both and proceed as follows:
 - 14.14.1 Pesticide and PCB – Refer to 14.8 for instructions on how to determine QC requirements. To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike. To all PCB QC, add 1.0mL of PCB 1660 LCS using a glass syringe dedicated for that particular spike.
 - 14.14.2 Pesticide only – To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike.

- 14.14.3 PCB only – To all PCB QC, add 1.0mL of PCB 1660 LCS with a glass syringe dedicated for that particular spike. 1660 is the standard PCB that we analyze for, if client specifies another PCB the extraction analyst will need to prepare another spike mix accordingly.
- 14.14.4 Enter the LIMS generated spike mix ID#, amount added, and the initials of the extraction and verifying analysts on the bench sheet and, later, in LIMS.
- 14.15 If the pH is not within 5.0-9.0 range, it must be adjusted using either the NaOH solution or Sulfuric Acid solution. If a pH adjustment is made, the details of the adjustment must be recorded in LIMS.
- 14.16 Seal and shake the separatory funnel vigorously for 3 minutes in the shaker apparatus with the stopcock open.
- 14.16.1 Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.
- 14.17 Allow the sample to set for a few minutes, if needed, after it has been shaken. It will separate into two layers with the solvent layer on the bottom.
- 14.17.1 If it forms an emulsion (thick, cloudy, viscous mixture that you cannot see through), drain what you believe to be 50mL into a 250mL centrifuge bottle.
- 14.17.2 Save and drain into this centrifuge bottle until the extraction is complete.
- 14.17.3 The emulsion must be centrifuged at 2500rpm for a good separation of the water from solvent.
- 14.18 Drain solvent layer into an appropriately labeled 250mL beaker.
- 14.19 Following steps 14.16 through 14.18, extract two more times with 40mL of methylene chloride combining all solvent extracts into the same appropriately labeled 250mL beaker.
- 14.20 Prepare a sample vial tray with 12mL vials and vial labels printed from LIMS. These labels contain the sample number, client name, initial/final volume, parameter, and date extracted.
- 14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by either or both of the following two methods.
- 14.21.1 Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not solvent. Discard this layer.
- 14.21.2 Use the smallest amount possible of Sodium Sulfate by sprinkling the top layer until it hardens, separates, and drops to the bottom.
- 14.22 Turbo-Vap Concentration
- 14.22.1 Rinse a Turbo-Vap tube and arrange it underneath a methylene chloride rinsed sodium sulfate filled filter funnel.
- 14.22.2 Using a sharpie, label the Turbo-Vap with the sample IDs
- 14.22.3 Pour the extract through the filter funnel into the appropriately labeled Turbo-Vap tube.
- 14.22.4 Rinse the beaker three times with methylene chloride and pour through funnel.
- 14.22.5 Rinse the filter funnel with methylene chloride once more and allow the funnels to sit until there is no more solvent dripping.
- 14.22.6 For solvent exchange purposes, add 50mL of hexane to each tube. Total volume in the Turbo-Vap tube should not exceed 200mL to avoid splattering

on the lid of the Turbo-Vap. If there is a large volume of methylene chloride extract, allow the sample to condense in Turbo-Vap until 75mL-100mL are left in the turbo tube.

- 14.22.7 Adjust pressure of nitrogen gas tank to >30psi, making sure that the tank has 200psi or more on the main valve.
- 14.22.8 Record the water bath temperature in the logbook located beside the TurboVap, making sure that it is 40°C-50°C.
- 14.22.9 Place turbo-vap tube in the Turbo-Vap. Be sure to push the tube down so the tip slides into the sensor well.
- 14.22.10 Close the lid and push corresponding well light to start concentration.
- 14.23 For PCBs Only – Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows:
 - 14.23.1 Add just enough methylene chloride to make the gel go back into solution
 - 14.23.2 Acid clean the extract and reconcentrate.
 - 14.23.3 Exchange with hexane again
 - 14.23.4 If gel forms again, add enough methylene chloride to get gel back into solution
 - 14.23.5 Transfer to a suitable container and record the final volume on the label and on bench sheet. Make sure to note the percentage of methylene chloride in sample.
- 14.24 When the samples reach a volume of 3mL-5mL, remove the tube from the batch
- 14.25 Hold the sample vial and tube in one hand at ~45° angle and 9” Pasteur pipette equipped with a latex bulb in the other.
- 14.26 Draw up sample and transfer into appropriately labeled 12mL sample vial. Be careful not to spill a drop during transfer.
- 14.27 Add 2-3mL of hexane to the tube and rinse several times using the pipette. Transfer this rinsate to sample vial and bring sample up to 10mL with hexane and cover the extract with a Teflon-sealed screw cap.
- 14.28 Take sample batch to GC Hobart sample refrigerator and log the sample numbers, analyst initials, and the date and time the samples were placed into the Hobart in the sample logbook located beside the refrigerator.
- 14.29 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations

Not applicable to this SOP

16.0 Method Performance

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not applicable to this SOP

19.0 Contingencies for Handling out-of-control or unacceptable data

Not applicable to this SOP

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition

21.2 40 CFR, Method 608

22.0 Tables, Diagrams, Flowcharts, and Validation Data

Not applicable to this SOP.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 338

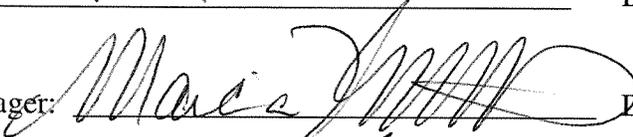
REVISION #: 08

EFFECTIVE DATE: 042910

**FLPRO
METHOD FOR DETERMINATION OF PETROLEUM RANGE
ORGANICS**

APPROVALS:

Lab Director:  Date: 4/29/10

Data Quality Manager:  Date: 4/29/10

Section Supervisor:  Date: 4/29/10

Changes Summary

Revision Date: 042910

- The SOP is a revision of rev07 dated 022410.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Table 2 has been updated to reflect method surrogate limits and in-house action limits for samples.

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PETROLEUM HYDROCARBONS

1. Test Method

1.1. This SOP is based upon method FL PRO.

2. Applicable Matrix

2.1. This SOP is applicable to the determination of the concentration of Petroleum Hydrocarbons in ground water, sediments, and wastes in the alkane range of C-8 to C-40.

3. Detection Limit

3.1. The detection limit for method FL-PRO is 0.085mg/L in water and 5.6 mg/Kg in soil.

4. Scope and Application

4.1. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

4.2. This method is recommended for use by experienced analysts or under the close supervision for such qualified personnel.

5. Summary of Record

5.1. Samples are extracted via proper extraction methods. A 1µL aliquot of the extract is injected into a GC system equipped with a flame ionization detector (FID). Quantification is based on the detector response in comparison to a series of alkane standards.

6. Definitions

6.1. Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2. Petroleum Hydrocarbons: All chromatographic peaks, both resolved and unresolved, eluting between the peak of n-octane (n-C8) and the peak end after n-tetracontane (n-C40). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from the FID response using baseline – baseline integration.

6.3. Petroleum Hydrocarbon Standard: A 17-component mix of all even-numbered alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

7. Interferences

7.1. All materials utilized during this analysis and the GC system must be demonstrated to be free from interference. Running frequent instrument blanks and methods blanks along with using pure, GC grade solvents will assist with the monitoring of interference's within the analytical system.

7.2. Any interference's co-extracted with the samples will vary considerably from source to source. Individual samples may require additional cleanup.

8. Health and Safety

- 8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.

9. Equipment and Supplies

- 9.1 Separatory Funnel - 2-Liter with Teflon stopcock
- 9.2 Beakers- 250 ml
- 9.3 Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- 9.4 Metal or wood rack - capable of holding at a minimum six glass evaporation tubes
- 9.5 Turbo-Vap Evaporator - heated and capable of temperature control ($\pm 5^{\circ}\text{C}$); the bath should be vented into a hood.
- 9.6 Silica Gel 60
- 9.7 Vials - 2 mL glass clear, with Teflon-lined screw cap
- 9.8 pH indicator paper - close range (0-6.0) and (7.0 - 14.0); wide range (1.0 - 12.0)
- 9.9 Syringe - 1000 μL
- 9.10 Graduated cylinder - Glass, Class A, 1000 mL
- 9.11 Pasteur pipette - length 9" and 5-3/4"
- 9.12 Pipette bulb
- 9.13 Aluminum foil - heavy duty
- 9.14 Nitrogen tank - equipped with pressure regulator
- 9.15 Ultrasonic Disrupter – capable of 300watts output, set on 10 Full power, pulse mode of 50%
- 9.16 A HP GC system, equipped with a flame ionization detector (FID), is used for analyzing extracts for all target analytes.
- 9.17 A Restek capillary column (RTX-5, 30m x 0.32mm x 0.25 μm) is used for analysis.
- 9.18 HP Chemstation Datasystem is used for data collection, detecting and storage.
- 9.19 Autosampler vials and caps appropriate to the sample tray are used for sample injection.
- 9.20 Microsyringes suitable for aliquoting 1.0 μL to 1000 μL s are used for standard preparation and sample dilution.
- 9.21 Class A volumetrics ranging from 1.0 ml to 250 mls are used for standard, spike and surrogate preparation.

10. Standards and Reagents

- 10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
 - 10.1.1. ASTM Type II Water.
 - 10.1.2. Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH into a 1L volumetric and cover with less than 1L reagent water. Use extreme caution when performing this step. Swirl the beaker until all pellets are dissolved (a stir plate can be used to mix the solution). This mixture gets very hot. Let stand until cool. Bring the solution up to the 1L mark with reagent water. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
 - 10.1.3. Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh placed in a Pyrex pan and heated at 400 $^{\circ}\text{C}$ overnight (minimum 4hrs), removed and cooled . Once cooled place in a labelled glass amber jar.
 - 10.1.4. Silica Gel 60 - Granular, anhydrous, trace pure 70-230 mesh. Weigh 60g in a 250 mL glass amber jar and add 1ml DI water to deactivate. Stored at room temperature.
 - 10.1.5. Glass Wool – Pre-rinse all glass wool used during the extraction process with Methylene Chloride.
 - 10.1.6. Sulfuric Acid Solution - (1:1), slowly add 500 mL of Sulfuric Acid to 500 mL of reagent water in a
 - 10.1.7. 1000 mL pyrex container. This mixture will get very warm. Allow to cool before use.

- 10.1.8. Extraction Solvent - Methylene Chloride (Dichloromethane (Please read SOP-336 before using this solvent in our laboratory)- Omnisolv - suitable for spectrophotometry and gas chromatography (JT Baker) or equivalent.
 - 10.1.9. Carbon Disulfide– (Omnisolv - suitable for spectrophotometry, liquid chromatography and gas chromatography (JT Baker) or equivalent.
 - 10.1.10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.
- 10.2. Stock Standards: Are non-Neat standards, received from vendors. These standards are used as intermediate standards to prepare working level standards. For unopened standards, if there is no expiration date assigned by the vendor, the expiration date must be assigned as 1 year from the date of receipt. For open stock standards, the expiration date is 6 months from the date the ampoule is opened or the vendor expiration date, whichever comes first. The following standards are used for the extraction and analysis of Petroleum Hydrocarbons:

<u>Vendor</u>	<u>Catalog #</u>	<u>Description/Conc</u>	<u>Used for Preparation of:</u>
Restek	31097	o-Terphenyl (OTP) 10,000 ug/ml	Curve & Surrogate Soln.
Restek	31096	2-Fluorobiphenyl (2FBP) 10,000 ug/ml	Curve & Surrogate Soln.
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc.=34,000ug/mL)	Spike Solution.
NSI	C-443-13	Florida TPH Mix 2000 ug/ml ea	Curve Solution
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc.=34,000ug/mL)	Second Source Standard

NOTE: The FL-PRO Petroleum Range Mix used for the preparation of the Spike Solution should always be a different Lot# from the Curve Standard.

10.3 Working Standards

10.3.1 Are standards made from Neat or from stock standards, and are intended for analytical runs. The expiration date for these standards is 6 months from the date of preparation or the expiration of the parent stock, whichever date is first.

10.3.2 Follow analytical judgement when using standards. Evaluate standards on a daily basis versus past standards and instrument performance. A standard may evaporate or breakdown if proper storage processes are not used. Therefore, standards may have to be discarded before expiration dates.

11. Sample Collections, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

12. Quality Control

12.1 An extraction batch must contain of no more than 20 client samples.

12.2 One BLK1, a BS1, BSD1, and a MS, MSD must be extracted in each batch.

12.3 Please follow guideline from Table 2 for meeting QC criteria.

12.4 All surrogates must pass the established laboratory criteria.

12.4.1 With samples requiring high level dilutions due to matrix interference or due to the abundance of target analytes, the surrogate will be diluted out and no recovery will be recorded. These samples can be reported.

12.4.2 For samples failing surrogate recovery high biased due to matrix interference, document the recoveries and notify the supervisor. In most cases, a Case Narrative should be filled out, the client should be notified, and the sample should be reported without a re-extraction. For samples failing the surrogate recovery (OTP) low biased, a re-extraction may need to be performed – check with supervisor. Any low recovery for surrogates reported to client must be noted in case narrative and a CAR must be filled out. This is on a case by case basis and at the discretion of the department supervisor.

13. Calibration and Standardization

13.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization. See section 14.3 of this SOP.

14. Procedure

14.1 Aqueous Extraction: All waters have a seven-day holding time. Determine the samples necessary to extract from the following sources. Note: never extract samples of unknown origin without discussion with supervisor):

14.1.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required

14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.

14.1.3 Periodically check LIMS throughout the day to determine what new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

14.1.4 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.

14.1.5 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid. Find out if any special dilutions are needed for the client. Routine procedures for difficult matrices are listed below.

14.1.6 BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made.

14.1.7 Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

14.1.8 Set up enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples.

14.1.9 Place an Avery label on each separatory funnel containing the Lab #.

14.1.10 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. The lab batch code is generated by LIMS. The BLK1 and BS1 label should include all lab #s in this set of samples.

14.1.11 Mark the amber glass container of each sample at the water meniscus with “white out” or with a sharpie for later determination of sample volume. Determine the initial pH of sample and record on extraction sheet. If needed, adjust pH to between 1.0 and 2.0.

14.1.12 ACID pH Adjusting: Adjust the pH to between 1.0 and 2.0, using 1:1 H₂SO₄. Add the acid solution to each sample, spike and method blank. Stopper and shake to insure that pH throughout

the sample is changed. Check the pH using a 9" pipette with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H₂SO₄ solution in small increments, as required to attain the proper pH. If sample is received without proper acid preservation, pH adjustment details must be recorded in LIMS.

- 14.1.13 Using the 1000-mL glass graduated cylinder measure 1000 mL of DI water and transfer it to a separatory funnel for each BLK1, BS1 & BSD1. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Rinse the sample bottle about 3-5 times with 10 mls aliquots of Methylene Chloride. Transfer this rinsate into the separatory funnel labeled with the sample ID.
- 14.1.14 Fill the sample bottle up to the mark with regular water. Now pour the water into a 1000 mL graduated cylinder. The volume measured is the initial volume to be documented for the sample in the LIMS.
Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.

NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.
- 14.1.15 Add 50 mL of Methylene Chloride to each sample and to all the batch QC. Shake the sep funnel twice and vent into the hood. Repeat this venting process 3-4 more times and then manually shake the sep funnel for two minutes. Vent the sep funnel at the end of the two minutes. Some samples may require additional venting due to excess pressure buildup. Please use precaution with highly volatile and reactive samples. Place sep funnel, inverted, in shaker apparatus with stopcock open for 3 minutes.
- 14.1.16 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. Drain the bottom organic layer into a labeled 250 ml glass beaker first passing the extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride.
- 14.1.17 Follow Steps 14.1.15 and 14.1.16, two more times with 40 mLs of methylene chloride using the automatic shaker. Collect the extract from this step into the same beaker.
- 14.1.18 Transfer the extract to a pre-rinsed zymark tube by first passing through a funnel with glass wool and baked sodium sulfate all pre-rinsed with methylene chloride. After pouring the extract into the zymark tube, rinse the collection beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.
- 14.1.19 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
- 14.1.20 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- 14.1.21 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.
- 14.1.22 Sign the batch into the extraction laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.
- 14.1.23 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

- 14.2 Solid Extraction (may also follow extraction procedure outlined in SOP 343). All solids have a fourteen-day holding time counted. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
- 14.2.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required
 - 14.2.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.
 - 14.2.3 Periodically check LIMS throughout the day to determine if new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
 - 14.2.5 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.
 - 14.2.6 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass jar. Routine procedures for difficult matrices are listed below:
 BAD MATRIX – for example a solid that is partially oil, see your supervisor to find out what dilution, if any should be made. Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.
 - 14.2.7 Get out enough 250mL beakers to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each extraction batch (up to a maximum of 20 samples). If sufficient sample is not available to perform a batch MS & MSD indicate such on the extraction sheet.
 - 14.2.8 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. Label each 250mL beaker with the Lab ID.
 - 14.2.9 Pre-weigh beakers and tare. Weigh 25g aliquot of the sample to the beaker and record weight to nearest 0.01g in extraction log. Add 25g dried Sodium Sulfate powder and stir the mixture well with a stainless steel spatula to a free-flowing sandy texture. If sample mixture forms large clumps, add more Sodium Sulfate to achieve proper texture (note in extraction log).
 - 14.2.10 *It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*
 - 14.2.10.1 *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
 - 14.2.10.2 *Two quarters should then be mixed to form halves.*
 - 14.2.10.3 *The two halves should be mixed to form a homogenous matrix. This procedure should be repeated several times until the sample is adequately mixed.*
 NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.
 - 14.2.11 Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.
 NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.
 - 14.2.12 Add 60 mL of Methylene Chloride to each sample and to all the batch QC. Sonicate each sample for 3 minutes in Ultrasonic Disruptor (set on 10 Full power – pulse mode) at a pulse rate of 50%.

- 14.2.13 Decant the Methylene Chloride extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride, into a rinsed zymark tube.
- 14.2.14 Follow Steps 14.2.12 and 14.2.13, one more time with 60 mLs of methylene chloride. Collect the extract from this step into the appropriately labeled tube.
- 14.2.15 After pouring the extract into the zymark tube, rinse the beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.
- 14.2.16 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Note the turbovap pressure and temperature on the extraction logbook.
- 14.2.17 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
- 14.2.18 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- 14.2.19 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.
- 14.2.20 Sign the batch into the GC laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.
- 14.2.21 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

14.3 GCFID Analysis

- 14.3.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.
- 14.3.2 Follow guidelines provided in the method for GC-FID conditions and sample volume to be injected for method FL Pro.
- 14.3.3 It is recommended that a solvent Blank be analyzed at the beginning of every sequence to ensure that the analytical instrument is free of contaminants.
- 14.3.4 All extracts within a batch are run on the Instrument after meeting calibration criteria as described in Table 2.
- 14.3.5 Qualitative and quantitative analysis is performed on samples.
 - 14.3.5.1 Qualitative Analysis for specific carbon ranges or fuel patterns, such as; mineral spirits, kerosene, JP-4 and heavy oils are performed per client request compared to specific standards. (See Table 3)
 - 14.3.5.2 Quantitative Analysis is performed using the following tools:
 - 14.3.5.2.1 Retention times for the range FL PRO C8-C40 are set daily using the mid-level of the calibration (if applicable) or the first CCV of the run by subtracting 0.05min. from the RT of C8 and adding 0.05min. to the RT of C40. FL PRO analysis is performed by running 6 calibration levels of a TPH mix from C-8 through C-40 (17 peaks). A response factor is calculated for each calibration standard (amount sum of 17 peaks/ std amount * 17), then an Average Response factor is calculated for all 6 standards. This Average Response Factor is put in the method for uncalibrated peaks. Percent RSD must be less than or equal to 20%.
 - 14.3.5.2.2 Surrogates o-Terphenyl and 2-fluorobiphenyl are added to each calibration standard at the same concentration. Initial calibration must pass acceptance criteria in Table 2.

14.3.5.2.3 Analyte concentration must be within the calibration curve range. If the analyte concentration exceeds the calibration curve range, the extract must be diluted & rerun to bring the concentration within the calibration range. Use the calculation in section 15 to report the final results for the sample.

14.3.5.2.4 Target analytes are calculated using the calibration curve and by incorporating any adjustments for initial or final volume and dilutions.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations throughout the laboratory.

15.2 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

The mean CF is calculated as follows:

15.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD \times 100}{\text{Avg}CF}$$

15.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) \times 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV } CF - \text{Average } CF) \times 100}{\text{Average } CF}$$

- 15.5 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve. The concentration of a specific analyte is calculated as follows:

Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = [(A_s) (V_t) (D)] / [(\overline{CF}) (V_i) (D)]$$

where:

A_s = Response for the analyte in the sample, units may be in area counts or peak height.

V_t = Total volume of sample, mL.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, $D = 1$.

\overline{CF} = Mean calibration factor from initial calibration (area/ng)

V_i = Volume of extract injected, μL .

V_s = Volume of aqueous sample, mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is $\mu\text{g/L}$.

Nonaqueous Samples:

$$\text{Concentration } (\mu\text{g/kg}) = [(A_s) (V_t) (D)] / [(CF) (V_i) (\overline{W_s})]$$

where:

W_s = Weight of dry sample extracted, g.

A_s , V_t , D , \overline{CF} and V_i have the same definition as for aqueous samples.

16. Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval.

16.2 See method FL-PRO for method performance.

17. Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, “Data Deviations/Interpretations/Exceptions: Laboratory Non-Conformance/ Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results,” provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. Waste Management and Pollution Prevention

- 20.1 Please see Waste Disposal SOPs 210 and 405 for proper waste disposal.
- 20.2 Quantity of chemicals purchased should be based on expected usage during it’s shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21. References

- 21.1 Method for Determination of Petroleum Range Organics (Method FL-PRO)

22. Tables, Diagrams, Flowcharts and Validation Data

Parameter	DL	LOD	LOQ/RL	Low Cal
FL-PRO	0.085ug/L	0.16ug/L	0.34ug/L	0.17ug/L
FL-PRO	5.6ug/Kg	10.7ug/Kg	22.6ug/Kg	11.3ug/Kg

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Calibration Curve	<ul style="list-style-type: none"> • Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at the RL/LOQ level 	<ul style="list-style-type: none"> • Linear correlation coefficient of at least 0.995 • Quadratic squared correlation coefficient of at least 0.99 • Average CF = < 20% RSD • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> • Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> • ≤ 25% drift or difference for all analytes 	<ul style="list-style-type: none"> • Re-analyze an ICV from a different source • Re-prepare and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> • At the beginning of every sequence • For every 10-client samples and at the end of the sequence • The concentration must be varied from low to mid range 	<ul style="list-style-type: none"> • ≤ 25% drift or difference for all analytes 	<ul style="list-style-type: none"> • Evaluate the system for required maintenance • Obtain passing CCV • Reanalyze all samples injected since last passing CCV • Q-qualify if reanalysis is not possible
MB	One per prep batch	<ul style="list-style-type: none"> • Must be less than ½ the RL/LOQ or <1/10th any sample concentration or <1/10th the regulatory limit – whichever is greater. 	<ul style="list-style-type: none"> • Re-analyze to confirm the positive value • If MB results are between the LOD and RL/LOQ, assess the data and notify the PM for possible further action • Re-extract affected samples associated with the MB • NCR and final report qualification will be required for affected samples if re-extraction is not possible.

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability									
Surrogates	Spike in every field or QC sample and standard	<table border="0"> <tr> <td>Surrogate</td> <td>Water</td> <td>Soil</td> </tr> <tr> <td>OTP</td> <td>82-142 (method) 30-140 (in-house)</td> <td>62-109 (method) 45-135 (in-house)</td> </tr> <tr> <td>2-FBP</td> <td>50-150 (default)</td> <td>50-150 (default)</td> </tr> </table> <p>Note: Project limits will be used when specified.</p>	Surrogate	Water	Soil	OTP	82-142 (method) 30-140 (in-house)	62-109 (method) 45-135 (in-house)	2-FBP	50-150 (default)	50-150 (default)	<ul style="list-style-type: none"> • Batch QC should pass method limits. • Reanalyze to confirm recovery if failing in-house limits. • Re-extract associated samples, if still failing in-house limits. • Q-qualify if re-extraction is not possible or verifies exceedence.
Surrogate	Water	Soil										
OTP	82-142 (method) 30-140 (in-house)	62-109 (method) 45-135 (in-house)										
2-FBP	50-150 (default)	50-150 (default)										
LCS	One per prep batch	Water 55-118% Soil 63-143% Note: Project limits will be used when specified.	<ul style="list-style-type: none"> • Reanalyze to confirm recovery. • Re-extract associated samples, if still failing • Q-qualify if re-extraction is not possible 									
LCSD	One per prep batch, when MS/MSD not included.	Water 55-118% RPD $\leq 20\%$ Soil 63-143% RPD $\leq 25\%$ Note: Project limits will be used when specified.	<ul style="list-style-type: none"> • See LCS 									
MS/MSD	One per prep batch, if sample volume available.	Water 41-110% RPD $\leq 20\%$ Soil 51-215% RPD $\leq 25\%$ Note: Project limits will be used when specified.	<ul style="list-style-type: none"> • Reanalyze to confirm recovery. • Re-extract if failure is judged to be due to extraction/analysis. • J-qualify associated parent sample if reporting from results exceeding limits. 									
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Must meet the criteria of the LCS for average recovery • Precision criteria is 20% standard deviation. 	<ul style="list-style-type: none"> • Re-prep and / or • Re-analysis 									
LOD Verification	Every quarter	<ul style="list-style-type: none"> • Parameter must be detected with response 3x the noise level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Raise concentration 									
LOQ Verification	Every quarter	<ul style="list-style-type: none"> • Bias Requirement: Organics 50-150% • The LOQ value must be greater than the LOD value 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Raise concentration 									

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Retention Time Study	<ul style="list-style-type: none"> • Prior to running samples • With major instrument changes 	3 injections over 72 hours – calculate standard deviation of the measured retention times. Windows +/- 3xSD	If <0.05minutes use +/0.05 minutes.

Table-3
Qualitative Analysis Tool

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 343 REVISION #: 01 EFFECTIVE DATE: 20100909

**BNA & Pesticide/PCBs & TPH NON-AQUEOUS MATRIX
(MICROWAVE EXTRACTION) USING SW-846 METHOD 3546**

APPROVALS:

Lab Director:  Date: 9/9/10

Data Quality Manager:  Date: 9/9/10

Section Supervisor:  Date: 9/9/10

Changes Summary

Revision 01, 09/09/2010

- SOP has been updated to reflect the correct QS SOPs and include missing solvent/spike information.

Revision 00, 08/01/09

- Review of SOP indicated no changes were necessary
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

**BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX
(Microwave Extraction)
Using SW846 METHOD 3546**

1. SCOPE AND APPLICATION

- a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

2. SUMMARY

- a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

3. INTERFERENCES

- a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
- b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
- c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

4. APPARATUS AND MATERIALS

- d. Stainless Steel spatula
- e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
- f. Microwave extraction Teflon tubes, capacity approximately 75mL
- g. Suitable Teflon cap and screw-top lid
- h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
- i. Vial – 2mL clear with Teflon-lined screw cap
- j. Vial – 12mL clear with Teflon-lined screw cap
- k. Syringe – 1mL, 500uL
- l. Pasteur pipet – 9” length
- m. Pasteur pipet bulb
- n. Labels – Dymo
- o. Aluminum foil – heavy duty
- p. Nitrogen tank – equipped with pressure regulator
- q. TurboVap Concentrator with 200mL concentrator tubes
- r. Teflon funnels for pouring off
- s. Balance – capable of weighing to 0.1grams
- t. Aluminum pie pans for mixing samples
- u. Filter paper – 185mm

5. REAGENTS

- a. Sodium Sulfate (Na_2SO_4) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
- b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
- c. Hexane – suitable for spectrophotometry and gas chromatography (Fisher #H303-4)
- d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
 - i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0ug/mL BN Surrogate spiking solution.)**
 - ii. **BNA Spiking Solution #1 & #2 (100 ug/mL)** – The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL PAH spiking solution.)** **The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
 - iii. **TCMX/DCB (2,4,5,6-Tetrachloro-metaxylene/Decachlorobiphenyl) Surrogate solution** is prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
 - iv. **PCB Spiking Solution** – Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
 - v. **Pesticide Spiking Solution** – A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
 - vi. **TPH Surrogate** – Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
 - vii. **TPH Spike** – A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

6. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
- b. Samples are preserved by cooling to 4°C.
- c. Holding time is 14 days from collection date to extraction.

7. PROCEDURE

- a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information. (DO NOT extract samples for which you have no information.):
 - i. Each day a backlog is generated in the LIMS providing all relevant sample information, including samples numbers and respective analysis required.
 - ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
 - iii. Check the backlog throughout the day to re-evaluate priority if needed.
- b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
- c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
- d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
- e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*

- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container

Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

- Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
- Quantitatively transfer samples to microwave tubes. Make sure samples are loaded in the rack in the order of the bench sheet.
- Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.
- Surrogate: **BNA** - using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **Pest/PCBs** - using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH – use the appropriate 1.0-mL glass syringe to add 1.0 mL of the appropriate surrogate to each sample, blank and spike.
- Spiking: For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
For Pest/PCB samples, determine if the sample will require a Pesticide

Spike and/or a PCB Spike. Proceed as follows:

Pesticide and PCB - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB.

For TPH - To the sample in each analytical batch selected for spiking, add 1mL of the appropriate spiking solution (i.e. DRO or TNEPH or MAEPH) using a 1.0 mL glass syringe dedicated to that spike.

- k. **Solvent:** Add 30mL methylene chloride for BNA/PAH/TPH extractions or 30ml hexane for Pest/PCB extractions.
- l. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
- m. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.
- n. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
- o. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

For 1-15 samples:

Max power: 800W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

For 16-40 samples:

Max power: 1600W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

- p. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
- q. Transfer the extract to a pre-rinsed turbo vap tube by first passing through

a funnel with P4 filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with the extraction solvent and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of the extraction solvent using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.

- r. Now concentrate the extract to 1.0mL using the turbovap concentrator.
 - i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45°C. The pressure target range should be about 20-25 psi.
 - ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
 - iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- s. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

8. DOCUMENTATION OF CAPABILITY (DOC)

- a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS08 for guidance.

9. WASTE MANAGEMENT AND POLLUTION PREVENTION

- a. Please see Waste Disposal SOP QS14 for the proper disposal of waste generated from this area.
- b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

10. METHOD PERFORMANCE

- a. Refer to SOP-201, SOP-211 and SOP-219 for method performance.

11. REFERENCES

- a. EPA Methods SW-846, Method 3546

12. DEFINITIONS

- a. Refer to SOP QS08 for definitions.

13. HEALTH AND SAFETY

- a. Wear appropriate personal protection equipment when working with chemicals or samples.
- b. Use the lab hoods when working with solvents.
- c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
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Marcia K. McGinnity
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In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Di-isopropyl ether
GC/MS	8260B	ETBE
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	t-Butyl alcohol
GC/MS	8260B	tert-Amyl methyl ether
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin

Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	3,5-Dinitroaniline
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	8011	1,2-Dibromoethane (EDB)
GC/ECD	8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7470A	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
IC	300.0	Chloride
IC	300.0	Fluoride

Non-Potable Water		
Technology	Method	Analyte
IC	300.0	Nitrate
IC	300.0	Nitrite
IC	300.0	Sulfate
IC	9056A	Chloride
IC	9056A	Fluoride
IC	9056A	Nitrate
IC	9056A	Nitrite
IC	9056A	Sulfate
Titration	SM 2320B 20th ed.	Alkalinity
ISE	SM 4500 B, D, 20th ed.	Ammonia
UV/Vis	7196A	Hexavalent Chromium
Colorimetric	353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th edition	Total Phosphorus
UV/Vis	SM 4500 PE, 20th edition	Ortho-Phosphorus
TOC	9060A/SM5310C, 20 th edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th edition	TDS
Colorimetric	9012A/B	Cyanide
Physical	1010A	Ignitability
Physical	9095B	Paint Filter
Probe	9040B/C	pH
Preparation	Method	Type
Preparation	1311	TCLP
Preparation	3005A	Metals digestion
Preparation	3010A	Metals digestion
Preparation	3510C	Organics Liquid Extraction
Preparation	5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBd)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7471A/B	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
UV/Vis	7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	9034	Sulfide
Probe	9045D	pH
Preparation	Method	Type
Preparation	1311	TCLP
Preparation	1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	3050B	Metals Digestion
Preparation	3546	Organics Microwave Extraction
Preparation	3541	Organics Soxhlet Extraction
Preparation	3550B	Organics Sonication
Preparation	SM 2540B 20th edition	Percent Solids (Percent Moisture)
Preparation	5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: _____



R. Douglas Leonard
Chief Technical Officer

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