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SAMPLING AND ANALYSIS PLAN (FIELD SAMPLING PLAN AND QUALITY ASSURANCE
PROJECT PLAN) FOR SITE ASSESSMENT SITES 4 AND 5 FUEL DEPOT WITH
TRANSMITTAL NS MAYPORT FL
8/17/2011
TETRA TECH

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001



Rev. 1
August 2011

Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan)

Site Assessment for U.S. Navy Fuel Depot – Sites 4 and 5

Navy Fuel Depot Jacksonville
Naval FISC Jacksonville
Jacksonville, Florida

Contract Task Order JM46

August 2011



NAS Jacksonville
Jacksonville, Florida 32212-0030



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Document Tracking Number 11JAX0182

August 17, 2011

Project Number 112G03309

Department of the Navy
Naval Facilities Southeast
ATTN: Mr. Brian Syme (OPDE 3)
Remedial Project Manager
135 Ajax Street North, Building 903
Naval Air Station Jacksonville
Jacksonville, FL 32212-0030

Reference: CLEAN Contract Number N62470-08-D-1001
Contract Task Order Number JM46

Subject: Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan), Site Assessment for U.S. Navy Fuel Depot – Sites 4 and 5, Navy Fuel Depot Jacksonville, Florida

Dear Mr. Syme:

Tetra Tech NUS, Inc. (Tetra Tech) is pleased to submit the Final Sampling and Analysis Plan for Site Assessment activities at the Navy Fuel Depot Sites 4 and 5, Jacksonville, Florida that was prepared for the United States Navy, Naval Facilities Engineering Command Southeast under Contract Task Order (CTO) JM46 for the Comprehensive Long-term Environmental Action Navy (CLEAN) Contract Number N62470-08-D-1001.

If you have any questions with regard to this submittal, please feel free to contact me at (904) 730-4669, extension 220, or via e-mail at Ben.Marshall@tetrattech.com.

Sincerely,

A handwritten signature in blue ink that reads "Ben Marshall".

Ben Marshall, P.E.
Task Order Manager

BM/lc

Enclosure

c: John Winters, FDEP (1 hardcopy, 1 CD)
Joe Marshall, Navy Fuel Depot (1 hardcopy, 1 CD)
Garth Glenn, Tetra Tech (letter only)
RDM (unbound, 1 CD)
Administrative Record (electronic only)
CTO JM46 Project File

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)
August 2011**

**Site Assessment
for
U.S. Navy Fuel Depot – Sites 4 and 5
Navy Fuel Depot Jacksonville
Jacksonville, Florida**

**Prepared for:
Naval Facilities Engineering Command Southeast
Naval FISC Jacksonville
Jacksonville, Florida 32212-0030**

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

**Prepared under:
Comprehensive Long-term Environmental Action Navy
CLEAN Contract No. N62470-08-D-1001
Contract Task Order JM46**

Project-Specific Sampling and Analysis Plan
Site Name/Project Name: US Navy Fuel Depot-Sites 4 and 5
Site Location: Navy Fuel Depot Jacksonville, FL

Site Assessment
Revision Number: 0
Revision Date: May 2011

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

Document Title: Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan),
Site Assessment for U.S. Navy Fuel Depot – Sites 4 and 5, Navy Fuel Depot
Jacksonville, Florida

Lead Organization: Naval Facilities Engineering Command Southeast (NAVFAC SE)

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

Preparer's Address and Telephone Number: 8640 Philips Highway , Suite 16
Jacksonville, Florida 32256
(904) 636-6125 Ext. 220

Preparation Date (Day/Month/Year): May 18, 2011

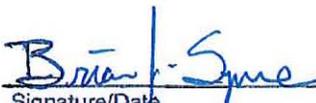
Investigative Organization's Project Manager:


Signature/Date
Ben Marshall, Tetra Tech NUS, Inc. 5-18-2011

Investigative Organization's Project QA Manager:


Signature/Date
Tom Johnston, PhD, Tetra Tech NUS, Inc. 5-18-2011

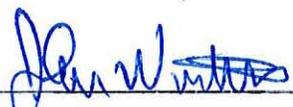
Lead Organization's Remedial Project Manager:


Signature/Date
Brian Syme, NAVFAC SE 13 June 2011

Lead Organization's QA Officer:


Digitally signed by
TUCKER.JONATHAN.P.1239524180
Date: 2011.06.08 14:54:31 -04'00'
Signature/Date
TBD, NAVFAC Atlantic Quality Assurance Officer
Chemist

Approval Signatures:


Signature/Date
John Winters, FDEP 8/16/2011

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) that encompasses Field Sampling Plan and Quality Assurance Project Plan requirements for a Site Assessment (SA) for U.S. Navy Fuel Depot - Sites 4 and 5 (Sites 4 and 5) at the Navy Fleet Industrial Supply Center (FISC) located in Jacksonville, Florida under the Comprehensive Long-term Environmental Action Navy (CLEAN) Contract No. N62467-08-D-1001, Contract Task Order JM46. The location of Sites 4 and 5 within the FISC Fuel Depot is presented on Figure ES-1.

This plan was generated for, and complies with, applicable United States Department of the Navy, United States Environmental Protection Agency (USEPA) Region 4, and Florida Department of Environmental Protection (FDEP) requirements, regulations, guidance, and technical standards. This includes the Department of Defense, Department of Energy, and USEPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. To comply with IDQTF requirements, this UFP-SAP is presented in the format of standard worksheets as specified in the Uniform Federal Policy for Quality Assurance Project Plans guidance documents (IDQTF, 2005).

This SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the planned investigation at the Fuel Depot. Protocols for sample collection, handling, and storage, chain-of-custody, laboratory and field analyses, data validation, and reporting are also addressed in this SAP.

Historical activities related to site operations at the Fuel Depot - Sites 4 and 5 may have led to contamination in surface soil, subsurface soil, and groundwater. Concentrations of Total Recoverable Petroleum Hydrocarbons (TRPH) measured in soil samples collected in 2007 from Sites 4 and 5 exceeded FDEP Soil Cleanup Target Levels (SCTLs) for petroleum related compounds.

The soil and groundwater investigation conducted in 2007 was limited in scope and the nature of potential contamination is unknown; thus, Tetra Tech was assigned the task of preparing this SAP for an SA of the soil and groundwater at the US Navy Fuel Depot - Sites 4 and 5. The SA will be conducted per FDEP 62-780 Florida Administrative Code (F.A.C.) requirements. The purpose of this SA is to obtain data that will enable the Partnering Team to (a) evaluate the data regarding the nature and extent of contamination at Sites 4 and 5 and to summarize findings and recommendations in a Site Assessment Report (SAR) and (b) decide if it is necessary to evaluate remedial alternatives in a Remedial Action Plan (RAP).

Both soil and groundwater samples will be collected and analyzed for volatile organic compounds, semivolatile organic compounds (including polycyclic aromatic hydrocarbons), polychlorinated biphenyls,

TRPH, speciated Total Petroleum Hydrocarbons, dioxins/furans (surface soil only), and metals. The reported concentrations of target analytes will be compared against the applicable FDEP Project Action Limits, which are conservative, risk-based screening criteria. Decisions related to any further action at the US Navy Fuel Depot - Sites 4 and 5 will be made by the Partnering Team based on the outcome of this investigation.

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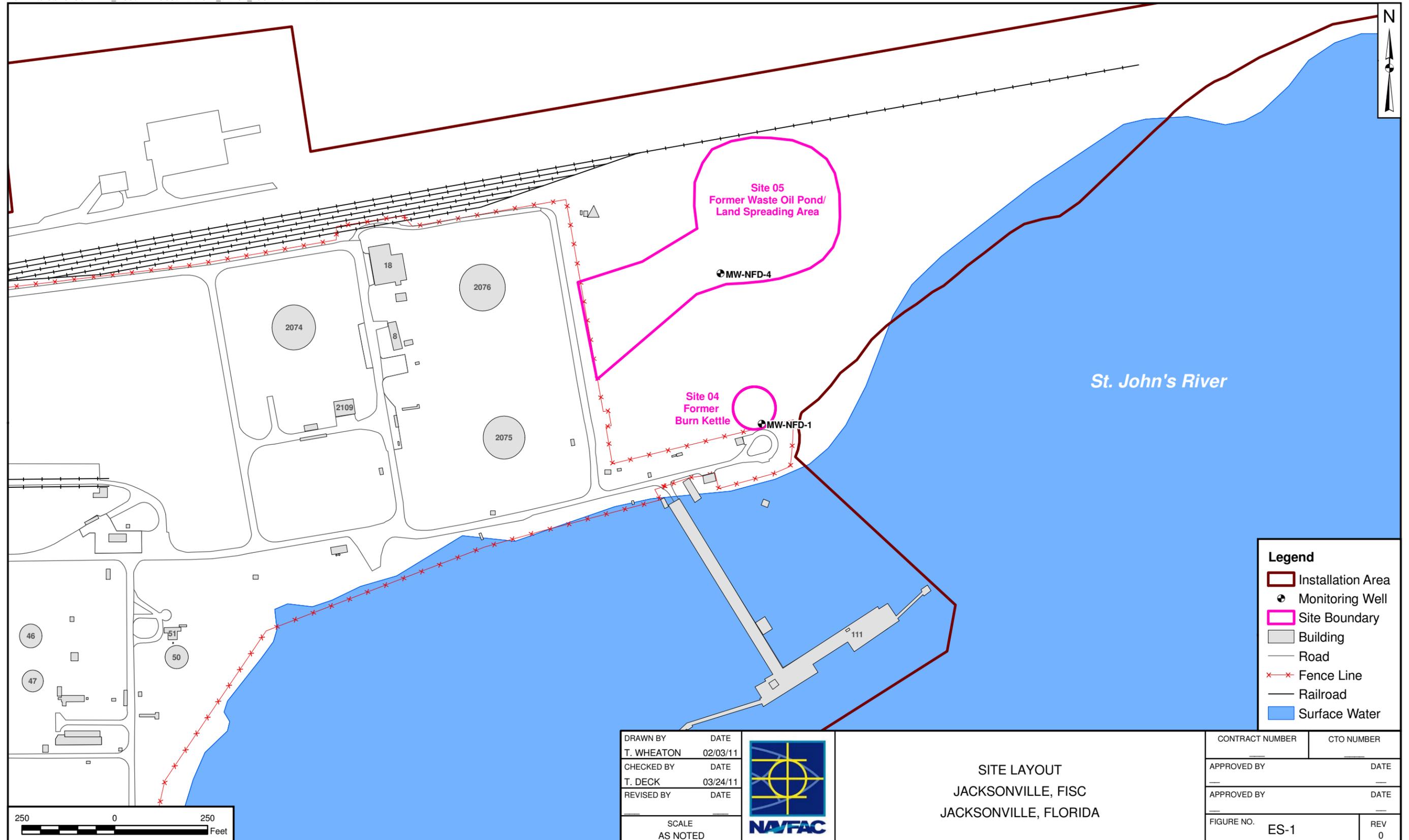


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APPENDICES

- A Field Standard Operating Procedures and Field Forms
- B Laboratory Standard Operating Procedures and Accreditations

ACRONYMS AND ABBREVIATIONS

°C	Degree Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
AES	Atomic Emission Spectroscopy
BFB	Bromofluorobenzene
bgs	below ground surface
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
CFA	Cape Fear Analytical, LLC
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Chain of Custody
COPC	Contaminant of Potential Concern
CPSM	Column Performance Check Solution
CSM	Conceptual Site Model
CTO	Contract Task Order
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
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
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EMPC	Estimated Maximum Positive Concentration
Empirical	Empirical Laboratories, LLC

ACRONYMS AND ABBREVIATIONS (CONTINUED)

Enco	Environmental Conservation Laboratories, Inc.
EPH	Extractable Petroleum Hydrocarbons
Ext.	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FDOH	Florida Department of Health
FID	Flame Ionization Detector
FISC	Fleet Industrial Supply Center
FL-PRO	Florida Residual Petroleum Organic Method
FOL	Field Operations Leader
FTMR	Field Task Modification Request
g	Gram
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
GCTL	Groundwater Cleanup Target Level
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HNO ₃	Nitric Acid
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
HSM	Health and Safety Manager
IAS	Initial Assessment Survey
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDQTF	Interagency Data Quality Task Force
IDW	Investigation-Derived Waste
IRP	Installation Restoration Program
IS	Internal Standard
JP-5	Jet Propellant Fuel No. 5
Katahdin	Katahdin Analytical Services, Inc.

ACRONYMS AND ABBREVIATIONS (CONTINUED)

L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
MADEP	Massachusetts Department of Environmental Protection
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
mg/kg	Milligram per Kilogram
mg/L	Milligram per Liter
mL	Milliliter
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NA	Not Applicable
NAD	North American Datum
NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast
NAVSTA	Naval Station
NC	No Criteria
NEDD	NIRIS Electronic Data Deliverable
NFA	No Further Action
NGVD	National Geodetic Vertical Datum
NIRIS	Naval Installation Restoration Information Solution
NTU	Nephelometric Turbidity Unit
OPR	Ongoing Precision Recovery
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
oz	Ounce
PAL	Project Action Limit
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit

ACRONYMS AND ABBREVIATIONS (CONTINUED)

PQLG	Practical Quantitation Limit Goal
PWD	Public Works Department
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
QSM	Quality Systems Manual for Environmental Laboratories
r	Linear Regression Correlation Coefficient
r ²	Coefficient of Determination
RAP	Remedial Action Plan
RBCA	Risk Based Corrective Action
RF	Response Factor
RMO	Risk Management Option
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSL	Regional Screening Level
SA	Site Assessment
SAR	Site Assessment Report
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SCTL	Soil Cleanup Target Level
SCTL-LCH	Soil Cleanup Target Level, Leachability
SCTL-RES	Soil Cleanup Target Level, Residential
SDG	Sample Delivery Group
SIM	Selected Ion Method
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language
SRCR	Sample Receipt Condition Report
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCL	Target Compound List
TCMX	Tetrachloro-m-xylene
TEL	Tetraethyl Lead

ACRONYMS AND ABBREVIATIONS (CONTINUED)

Tetra Tech	Tetra Tech NUS, Inc.
TRPH	Total Recoverable Petroleum Hydrocarbons
TPH	Total Petroleum Hydrocarbons
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
µg/L	Microgram per Liter
USEPA	United States Environmental Protection Agency
USEPA-RSL	United States Environmental Protection Agency Regional Screening Level
USGS	United States Geological Survey
VOC	Volatile Organic Compound
VPH	Volatile Petroleum Hydrocarbon

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: U.S. Navy Fuel Depot - Sites 4 and 5
Contractor Name: Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number: N62470-08-D-1001
Contract Title: Comprehensive Long-term Environmental Action Navy (CLEAN)
Work Assignment Number: Contract Task Order (CTO) JM46

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

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), as reauthorized by Superfund Amendments and Reauthorization Act (SARA) and the Florida Department of Environmental Protection (FDEP) Division of Waste under 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) Scoping Meetings (Naval Station [NAVSTA] Mayport Partnering Team)	February 16, 2011

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

Title	Date
None	None

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

Naval Facilities Engineering Command Southeast (NAVFAC SE)
US Navy (NAVSTA Mayport) (property owner)
FDEP (regulatory stakeholder)

7. Lead organization

Naval Facilities Engineering Command Southeast (NAVFAC SE)

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

Not Applicable (NA), as there are no exclusions.

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

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Brian Syme	Navy Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC SE OPDE3 PO Box 30 Bldg 903 NAS Jacksonville, FL 32212	(904) 542-6151	brian.syme1@navy.mil
Joe Marshall	Installation Restoration Program (IRP) Manager/ Navy Fuel Depot Jacksonville Point of Contact (POC)	US Navy Fuel Depot - Jacksonville 8808 Somers Road Jacksonville, FL 32226	(904) 696-6556 Ext. 202	joseph.r.marshall.ctr@navy.mil
To Be Determined (TBD)	NAVFAC Quality Assurance Officer (QAO)/ Government Chemist	TBD	TBD	TBD
John Winters	FDEP RPM/ Provides Regulator Input	FDEP 2600 Blair Stone Road, MS 4500 Tallahassee, FL 32399-2400	(850) 245-8999	john.winters@dep.state.fl.us
Ben Marshall	Project Manager (PMPM)/ Manages Project Activities	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Extension (Ext.) 220	ben.marshall@tetrattech.com
Kevin Weichert	Field Operations Leader (FOL) / Site Safety Officer (SSO)/ Manages Field Operation and Site Safety Issues	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Ext. 219	Kevin.Weichert@tetrattech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Tom Johnston, PhD (electronic copy only)	Quality Assurance Manager (QAM)/ Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8615	tom.johnston@tetrattech.com
Mark Peterson (electronic copy only)	FL Operations Manager/ Provides DQO and SAP Support	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Ext. 213	Mark.peterson@tetrattech.com
Tom Dickson [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) 921-8457	tom.dickson@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)	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
Julie Johnson	Administrative Project Assistant (Jacksonville Administrative Record)	Tetra Tech 8640 Philips Hwy, Suite 16 Jacksonville, FL 32256	(904) 730-4669 Ext. 224	julie.johnson@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

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Kelly Perkins (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Katahdin Analytical Services, Inc. (Katahdin) 600 Technology Way Scarborough, ME 04074	(207) 874-2400 Ext 17	kperkins@katahdin.com
Chris Cornwell (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Cape Fear Analytical, LLC (CFA) 3306 Kitty Hawk Rd. Suite 120 Wilmington, NC 28405	(910) 795-0421	chris.cornwell@cfanalytical.com
Marcia Colon (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Environmental Conservation Laboratories, Inc. (Enco) 10775 Central Port Drive Orlando, Florida 32824	(407) 826-5314	mcolon@encolabs.com
Well Installation and Direct-Push Technology (DPT) Driller(s) (TBD) (electronic copy only)	Subcontractor PM/ Provides Drilling Services	TBD	TBD	TBD
Utility Locator (electronic copy only)	Subcontractor PM/ Provides Utility Locating Services	TBD	TBD	TBD

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 and are listed in Worksheet #29 as project records.
2. E-mails will be sent to the Navy, Tetra Tech, and subcontractor project personnel who will be requested to verify by e-mail that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and is identified in Worksheet #29.

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

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Partnering Team Personnel					
Brian Syme	Navy/ RPM/ Manages Project Activities for the Navy	(904) 542-6151	See Worksheet #1 for signature	All	
Joe Marshall	Navy/ IRP Manager/ Navy Fuel Depot Jacksonville POC	(904) 696-6556 Ext. 202		All	
John Winters	FDEP/ RPM/ Provides Regulator Input	(850) 245-8999	See Worksheet #1 for signature	All	

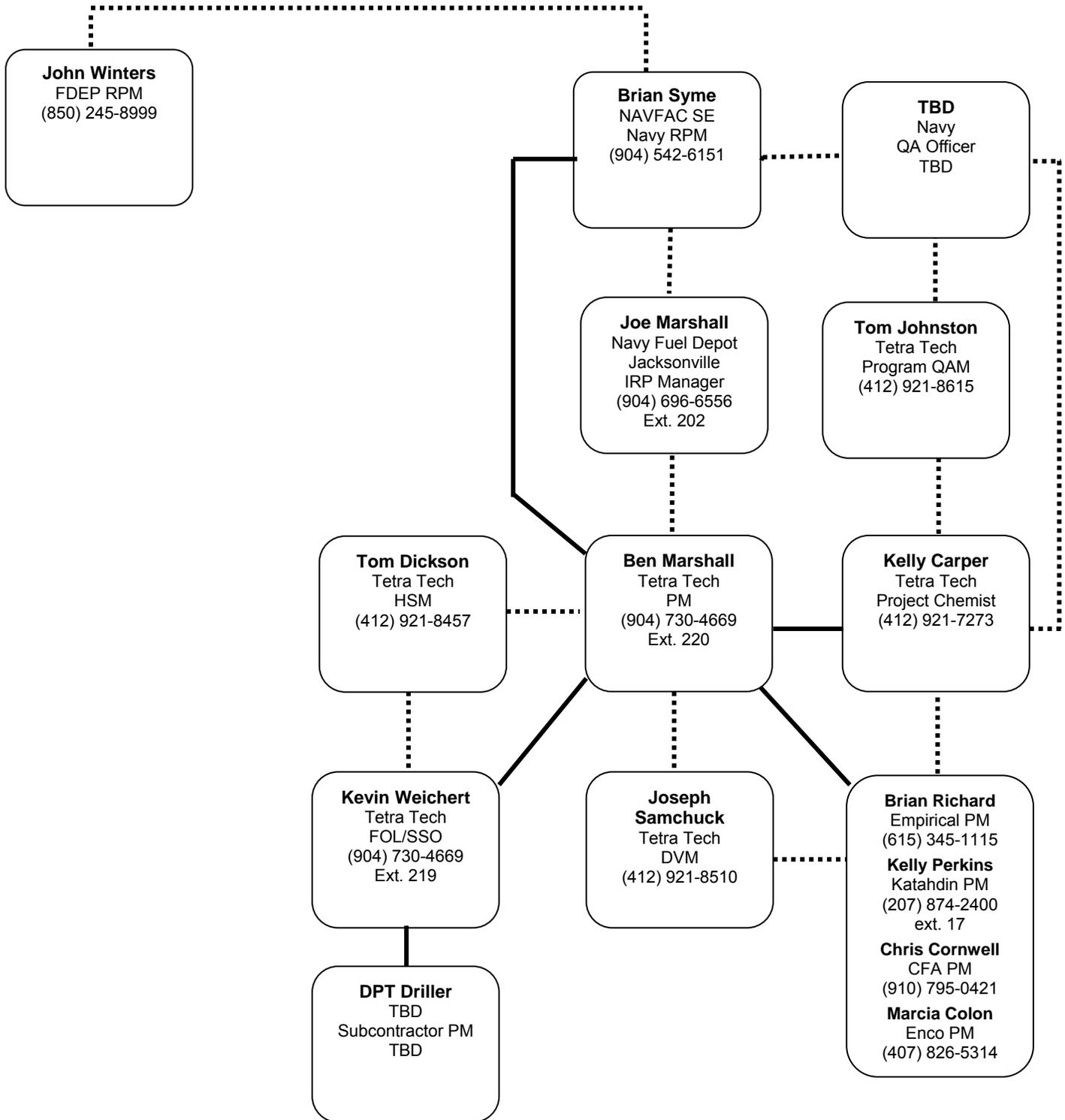
Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Tetra Tech Partnering Team Personnel					
Ben Marshall	Tetra Tech/ PM/ Manages Project Activities	(904) 730-4669 Ext. 220	See Worksheet #1 for signature	All	
Kevin Weichert	Tetra Tech/ FOL/SSO/ Manages Field Operation and Site Safety Issues	(904) 730-4669 Ext. 219		All	
Tom Johnston	Tetra Tech/ QAM/ Manages NAVFAC SE Contract QA Program and Implementation	(412) 921-8615	See Worksheet #1 for signature	All	
Matt Soltis	Tetra Tech/ HSM/ Manages Corporate Health and Safety Program	(412) 921-8912	See HASP for signature	HASP	
Thomas Deck	Tetra Tech/ Environmental Scientist/ Provides DQO and SAP Support	(904) 730-4669 Ext. 228		All	
Kelly Carper	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	(412) 921-7273		All	
Joseph Samchuck	Tetra Tech/ DVM/ Manages Data Validation	(412) 921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
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	
Kelly Perkins	Katahdin/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(207) 874-2400 Ext. 17		Worksheets #6, #12, #14, #15, #19, #23- 28, #30, and #34-36	
Chris Cornwell	CFA PM/ Representative for Laboratory and Analytical Issues	(910) 795-0421		Worksheets #6, #12, #14, #15, #19, #23- 28, #30, and #34-36	
Marcia Colon	Enco/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(407) 826-5314		Worksheets #6, #12, #14, #15, #19, #23- 28, #30, and #34-36	
Driller(s) - TBD	TBD/ DPT and Well Installation Subcontractor PM/ Provides Drilling Services	TBD		Worksheets #6, #14, #17, and Figures	
Utility Locator - TBD	TBD/ Utility Locator Subcontractor PM/ Provides Utility Locating Services	TBD		Worksheets #6, #14, #17, and Figures	

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

Lines of Authority —————

..... Lines of Communication



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

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
SAP amendments	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM	Kevin Weichert Ben Marshall Brian Syme	(904) 730-4669 Ext. 219 (904) 730-4669 Ext. 220 (904) 542-6151	<p>The Tetra Tech FOL will verbally inform the Tetra Tech PM within 24 hours of realizing a need for an amendment.</p> <p>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.</p> <p>SAP amendments will be submitted by the Tetra Tech PM to the Navy RPM for review and approval. The Navy RPM will notify the regulators by mail of changes to the SAP.</p> <p>The Tetra Tech PM will send scope changes to the Partnering Team via e-mail within 1 business day.</p>
Changes in schedule	Tetra Tech PM Navy RPM Navy Fuel Depot Jacksonville POC	Ben Marshall Brian Syme Joe Marshall	(904) 730-4669 Ext. 220 (904) 542-6151 (904) 696-6556 Ext. 202	The Tetra Tech PM will verbally inform the Navy RPM and the Navy Fuel Depot Jacksonville POC on the day that schedule change is known and document via schedule impact letter within 1 business day of when impact is realized.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
Issues in the field that lead to changes in the scope of work	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM Navy Fuel Depot Jacksonville POC	Kevin Weichert Ben Marshall Brian Syme Joe Marshall	(904) 730-4669 Ext. 219 (904) 730-4669 Ext. 220 (904) 542-6151 (904) 696-6556 Ext.202	<p>The Tetra Tech FOL will verbally inform the Tetra Tech PM on the day that the issue is discovered.</p> <p>The Tetra Tech PM will inform the Navy RPM and the Navy Fuel Depot Jacksonville POC (verbally or via e-mail) within 1 business day of discovery.</p> <p>The Navy RPM will issue scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed.</p> <p>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.</p>
Recommendation to stop work and initiate work upon corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Tetra Tech HSM Tetra Tech Project Chemist Navy RPM Navy Fuel Depot Jacksonville POC	Kevin Weichert Ben Marshall Tom Johnston Tom Dickson Kelly Carper Brian Syme Joe Marshall	(904) 730-4669 Ext. 219 (904) 730-4669 Ext. 220 (412) 921-8615 (412) 921-8457 (412) 921-7273 (904) 542-6151 (904) 696-6556 Ext.202	<p>If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform on-site personnel, subcontractor(s), the Navy Fuel Depot Jacksonville POC, and the identified Partnering Team members within 1 hour (verbally or by e-mail).</p> <p>If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.</p>
Corrective action for field program	Tetra Tech QAM Tetra Tech PM Navy RPM	Tom Johnston Ben Marshall Brian Syme	(412) 921-8615 (904) 730-4669 Ext. 220 (904) 542-6151	<p>The Tetra Tech QAM will notify the Tetra Tech PM verbally or by e-mail within 1 business day that the corrective action has been completed.</p> <p>The Tetra Tech PM will then notify the Navy RPM (verbally or by e-mail) within 1 business day.</p>
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech TOM	Kevin Weichert Ben Marshall	(904) 730-4669 Ext. 219 (904) 730-4669 Ext. 220	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 Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
Analytical data quality issues	Laboratory PM Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Brian Richard Kelly Richards Chris Cornwell Marcia Colon Kelly Carper Ben Marshall Brian Syme	(615) 345-1115 (207) 874-2400 ext. 17 (910) 795-0421 (904) 296-3007 (412) 921-7273 (904) 730-4669 Ext. 220 (904) 542-6151	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within 1 business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech PM within 1 business day. 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. When the Navy RPM is informed of the issue, the Navy RPM should inform the Navy QAO if it is a significant analytical issue.

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

The personnel responsible for implementing the SAP are identified in the following table. Resumes are available upon request.

Name	Title/Role	Organizational Affiliation	Responsibilities
Brian Syme	Navy RPM/ Manages project activities for the Navy	NAVFAC SE	Oversees project implementation including scoping, data review, and evaluation.
Joe Marshall	IRP Manager/ Manages daily site activities related to this project	Navy Fuel Depot Jacksonville	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
John Winters	RPM/ Provides regulator input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP.
Ben Marshall	PM/ Manages project on a daily basis	Tetra Tech	Oversees project and manages financial, schedule, and technical day-to-day activities of the project.
Kevin Weichert	FOL/SSO Manages field operations and oversees site activities to ensure safety requirements are met	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities. As the SSO, is responsible for on-site project-specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the 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 CLEAN Program 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.
Kelly Perkins	Laboratory PM/ Representative for laboratory and analytical issues	Katahdin	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
Chris Cornwell	Laboratory PM/ Representative for laboratory and analytical issues	CFA	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
Marcia Colon	Laboratory PM/ Representative for laboratory and analytical issues	Enco	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
TBD	Driller(s)/ Provides drilling services	TBD	Performs DPT soil borings according to scope of work.
TBD	Utility Locator/ Provides utility locating services	TBD	Performs utility location.

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 will be required to have completed appropriate Hazardous Waste Operations and Emergency Response (HAZWOPER) training specified in Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 (e). Project-specific safety requirements are addressed in greater detail in the site-specific HASP.

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

Project Name: US Navy Fuel Depot Projected Date(s) of Sampling: August, September, November 2011 Project Manager: Ben Marshall		Site Name: US Navy Fuel Depot – Sites 4 and 5 Site Location: FISC Navy Fuel Depot, Jacksonville, Florida			
Date of Session: February 16, 2011 Scoping Session Purpose: DQO Development					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Brian Syme	Navy RPM	NAVFAC SE	(904) 542-6151	Brian.Syme1@navy.mil	Navy RPM
Joe Marshall	IRP Manager Navy Fuel Depot Jacksonville	NAVFAC SE	(904) 696-6556 Ext. 202	joseph.r.marshall.ctr@navy.mil	IRP Manager
John Winters	Federal Facilities RPM	FDEP	(850) 245-8999	John.Winters@dep.state.fl.us	FDEP RPM
Ben Marshall	PM	Tetra Tech	(904) 730-4669 Ext. 220	Ben.Marshall@tetrattech.com	PM
Mark Peterson	Florida Operations Manager	Tetra Tech	(904)730-4669 Ext. 213	Mark.Peterson@tetrattech.com	Technical Support: Assessment, Regulatory Compliance, and Remediation
Thomas Deck	DQO Support	Tetra Tech	(904) 730-4669 Ext. 228	Tom.Deck@tetrattech.com	Project Support
Libby Claggett	Administrative Project Assistant I	Tetra Tech	(904) 730-4669	Libby.Claggett@tetrattech.com	Scribe

Comments/Decisions:

The Site background and the DQO process were presented and the following was discussed for Sites 4 and 5:

Joe Marshall confirmed that the burn kettle at Site 4 was removed and recycled in 2010, and that the burn kettle was full of a thick, black substance with a petroleum odor. The sludge was sampled and contained some low level dioxins. Monitoring wells NFD-2 and NFD-3 are believed to have been destroyed.

The project goals include site assessments for Sites 4 and 5. John confirmed that Sites 4 and 5 are Chapter 62-780 F.A.C. sites (and not Chapter 62-770).

The site assessment approach will be completed in phases. Phase I will determine contaminants of potential concern (COPCs) for Sites 4 and 5 by sampling soil and groundwater in the areas that indicated a release. Phase II will determine the lateral and vertical extent of soil and groundwater impacts within the facility boundaries. Phase III, if necessary, will include limited soil and groundwater sampling to address any data gaps in the site assessments. John Winters stated that step out samples (Phase II) need to include both vertical and horizontal boundary locations.

If fill material at the site came from NAVSTA Mayport, the arsenic background value of 13.7 mg/kg could apply.

Decisions

Partnering team agreed on sampling approach as described in Worksheet # 17

Partnering team agreed on parameters as described in Worksheet #15

Partnering team agreed on DQOs and decision rules as described in Worksheet #10 and Worksheet #11.

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

10.1 INTRODUCTION

Site 4, Slurry Burn Pit and Burn Kettle and Site 5, Old Oil Pond and Land Spreading Area are located at the Fleet and Industrial Supply Center (FISC) Navy Fuel Depot. The FISC Navy Fuel Depot is located along the north bank of the St. Johns River in Jacksonville, Florida (Figure 10-1) and the area of Sites 4 and 5 is approximately 4 acres. The facility supports NAVSTA Mayport located to the east and Naval Air Station (NAS) Jacksonville located to the south. Additional details concerning the Conceptual Site Model (CSM), including historical site activities, past investigations, and current site conditions are provided below.

10.2 SITE DESCRIPTION

Site 4: Slurry Burn Pit & Burn Kettle

Site 4 is approximately 1,500 square feet and is located on the FISC Navy Fuel Depot east of Fifth Street and Tank #2075. The St. Johns River is located to the south and the east of Site 4. Site activities included the disposal and burning of off grade petroleum products in the steel kettle and slurry pit. Station generated trash was also disposed of at this site and routinely burned in the burn kettle. This practice was conducted from approximately 1965 to 1967. According to the Facility Manager, the burn kettle was removed and the associated kettle pipe was abandoned in place in 2010. The abandoned pipe runs south where it surfaces approximately 82 feet from the former location of the Burn Kettle.

Site 5: Old Oil Pond and Land Spreading Area

Site 5 is approximately 3.5 acres and is located on the FISC Navy Fuel Depot approximately 300 feet to the north of Site 4. The Old Oil Pond area was excavated and diked in the 1950s prior to use. In 1971, the area was re-graded using oil/sludge contaminated soils. Visual inspection indicates that the vegetation is stressed at the site with surface soil indicating a hard crust consisting of a mix of sand and tar-like substance. The surface soil impact was observed intermittently over an area covering approximately 160,000 square feet.

An aerial photograph of Sites 4 and 5 that shows the site features and the surrounding area is presented on Figure 10-2.

10.3 PREVIOUS INVESTIGATIONS AND ACTIONS

Previous investigations at Sites 4 and 5 include the Initial Assessment Survey (IAS) in 1983, a Confirmation Study in 1983 (Site 5), a Characterization Phase of the Confirmation Study in 1986 (Site 5), and an Initial Site Investigation in 2007 (Sites 4 and 5). Some of these investigations included only a review of available files on the sites and a determination if there was a possibility of contaminant release to the environment based on the information in the files and some of the investigations included field activities such as visual inspections, soil, and groundwater sampling. The current investigation is being conducted in accordance with FDEP Site Assessment Report (SAR) guidelines established in Chapter 62-780, F.A.C.

Initial Assessment Survey, March 1983

In March 1983, an IAS was conducted at the FISC Navy Fuel Depot. The IAS included record searches and interviews that identified five sites that were potentially contaminated. Included in these five sites were Sites 4 and 5.

During the IAS it was identified that approximately 336,000 gallons of Navy special fuel and water from off-grade petroleum products, 840 gallons of tetraethyl lead (TEL) contaminated sludge and rust from aviation gasoline storage tanks, and an unknown quantity of trash, cans, paper, etc. from the Navy Fuel Depot were disposed of at Site 4. At Site 5, approximately 336,000 gallons of contaminated fuel oil from an aircraft carrier were disposed of with garbage and wood through 1967. Approximately 5,000 gallons of JP-5 fuel mixed with TEL sludge from cleaning tanks was disposed of prior to 1967. It was recommended in the IAS Report that a Confirmation Study be conducted.

Confirmation Study (Site 5), August 1983

During the Confirmation Study conducted in August 1983, 32 borings were sampled for soil to determine the areal extent of contamination at Site 5. Of these borings, two were submitted for laboratory analysis. Additionally, two monitoring wells (NFD-1 and NFD-2) were installed on the eastern and southern edge of Site 5 and samples were collected and analyzed for pH, specific conductance, volatile organic compounds (VOCs), cyanide, and metals. The results of the investigation indicated that soil at Site 5 contained non-hazardous waste. Results from the monitoring wells indicated the presence of VOC contamination at low levels. It was recommended that additional monitoring wells be installed to further investigate potential groundwater contamination at the site.

Characterization Phase of the Confirmation Study (Site 5), February 1986

During the Characterization Phase of the Confirmation Study conducted in February 1986, two monitoring wells were installed south of the location of the 32 soil borings collected in August 1983. Samples were

collected from monitoring wells NFD-2, NFD-3, and NFD-4 and were analyzed for dissolved aromatic hydrocarbons and petroleum hydrocarbons. Petroleum hydrocarbons were detected in samples from two groundwater wells at 1 milligram per liter (mg/L). Well locations NFD-1 and NFD-4 (NFD-2 and NFD-3 were not located during the last site inspection) are presented in Figure 17-1.

Initial Site Investigation, June 2007

During the initial site investigation conducted in June 2007 soil was collected at Sites 4 and 5. At Site 4, two soil samples were collected surrounding the burn kettle. Additionally, field observations at Site 4 indicated that a release of heavy petroleum products and burned material occurred which had left a hard crust on the land surface in the vicinity of the burn kettle. Results from the soil samples included concentrations of Total Recoverable Petroleum Hydrocarbons (TRPH) at 3,860 milligrams per kilogram (mg/kg) and 6,610 mg/kg exceeding the FDEP residential Soil Cleanup Target Levels (SCTL) of 460 mg/kg. Soil was screened from four boring locations from 0 to 6 inches and at 2 feet with a flame ionization detector (FID) around the kettle with all readings being well below any action level. At Site 5, visual inspection indicated that the vegetation was stressed, with areas of soil having a hard crust consisting of a mix of sand and tar-like substance. The visual inspection indicated the area of impacted soil (containing hard crusted surface) is spread over an area of approximately 160,000 square feet. Based on the results of the visual inspection, one soil sample was collected in an area with heavily crusted soils from 0 to 6 inches. The soil had a measured TRPH concentration of 16,900 mg/kg, exceeding the FDEP residential SCTL.

10.4 CONCEPTUAL SITE MODEL

The CSM for Sites 4 and 5 is described in the following sections and is visually depicted on Figure 10-3. The purpose of this investigation is to collect data to define the nature and extent of contamination at Sites 4 and 5. As described in the following sections, there is limited historical data and significant uncertainty remains about the nature and extent of potential contamination that may exist at Sites 4 and 5.

10.4.1 Geology and Hydrogeology

Groundwater underneath the FISC Navy Fuel Depot is present in three main aquifer systems. In general, the surficial, shallow, or upper aquifer ranges from 10 to 90 feet in thickness and contains unconsolidated sediments that include clay, silt, sands, shelly sands, and shell beds. Documents suggest that the top of the surficial aquifer in some areas of FISC Navy Fuel Depot is 3 to 8 feet below ground surface (bgs), whereas the top of the surficial aquifer in other areas are approximately 15 to 35 feet bgs. At Sites 4 and 5, the top of the surficial aquifer is estimated to be approximately 3 feet bgs based on previous investigation groundwater measurements and proximity to the St. Johns River. Generally, groundwater in

the surficial aquifer flows from high topography to low topography and toward the St. Johns River. According to the United States Geological Survey (USGS), groundwater in the surficial aquifer is not a water supply for FISC Navy Fuel Depot or the surrounding areas. The intermediate Hawthorne aquifer underlying the FISC Navy Fuel Depot vicinity ranges from about 35 to 400 feet bgs and the Floridan aquifer begins about 400 feet bgs and contains more than 1,000 feet of dolostone formations and limestone.

Surface water at FISC Navy Fuel Depot is primarily the St. Johns River, which forms the southern and eastern boundary of the facility. Stormwater run-off at Sites 4 and 5 drains toward and into the St. Johns River.

10.4.2 Potential or Known Sources of Contamination

Site 4: Slurry Burn Pit & Burn Kettle

Diesel fuel and bottom sludge from tank cleaning wash water in addition to activities associated with the burn kettle may have led to the contamination in surface soil, subsurface soil, and groundwater (see Figure 10-3). Chemical concentrations of TRPH were detected during a previous investigation in two surface soil samples collected at Site 4. New data is required to determine the nature and extent of contamination at the slurry burn pit, burn kettle, and associated piping at Site 4. The new data set should be robust enough to spatially represent the entire slurry burn pit and burn kettle area.

Diesel fuel and bottom sludge from tank cleaning wash water in addition to activities associated with the burn kettle are the likely source of any potential contamination at Site 4. Results of the IAS, Confirmation Study, and Initial Site Investigation including limited analytical results and visual inspection indicate that releases from facility activities may have occurred directly onto the surface soil and that the releases potentially have migrated into site subsurface soil and groundwater.

Following review of historical investigations, the Partnering Team agreed that the target analytes for Site 4 will include VOCs; semivolatile organic compounds (SVOCs), including polycyclic aromatic hydrocarbons (PAHs); polychlorinated biphenyls (PCBs); metals; dioxins/furans (surface soil only); TRPH; and speciated Total Petroleum Hydrocarbons (TPH), including Volatile Petroleum Hydrocarbons (VPH) and Extractable Petroleum Hydrocarbons (EPH) (see Worksheet #15).

Site 5: Old Oil Pond and Land Spreading Area

Historical disposal of fuel oil and JP-5 mixed with TEL-contaminated sludge may have led to the contamination in surface soil, subsurface soil, and groundwater (see Figure 10-3) at Site 5. Chemical concentrations of TRPH were detected during a previous investigation in one surface soil sample collected at Site 5. New data is required to determine the nature and extent of contamination at the old oil

pond and land spreading area at Site 5. The new data set should be robust enough to spatially represent the old oil pond and land spreading area.

Fuel oil and JP-5 mixed with TEL-contaminated sludge is the likely source of any contamination at Site 5. Results of the IAS, Confirmation Study, and Initial Site Investigation including limited analytical results and visual inspection indicate that releases from facility activities may have occurred directly onto the surface soil and potentially have migrated into site subsurface soil and groundwater.

Following review of historical investigations, the NAVSTA Mayport Partnering Team agreed that the target analytes for Site 5 will include VOCs, SVOCs (including PAHs), PCBs, metals, dioxins/furans (surface soil only), TRPH, and speciated TPH (see Worksheet #15).

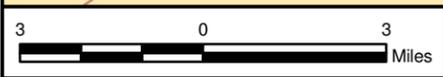
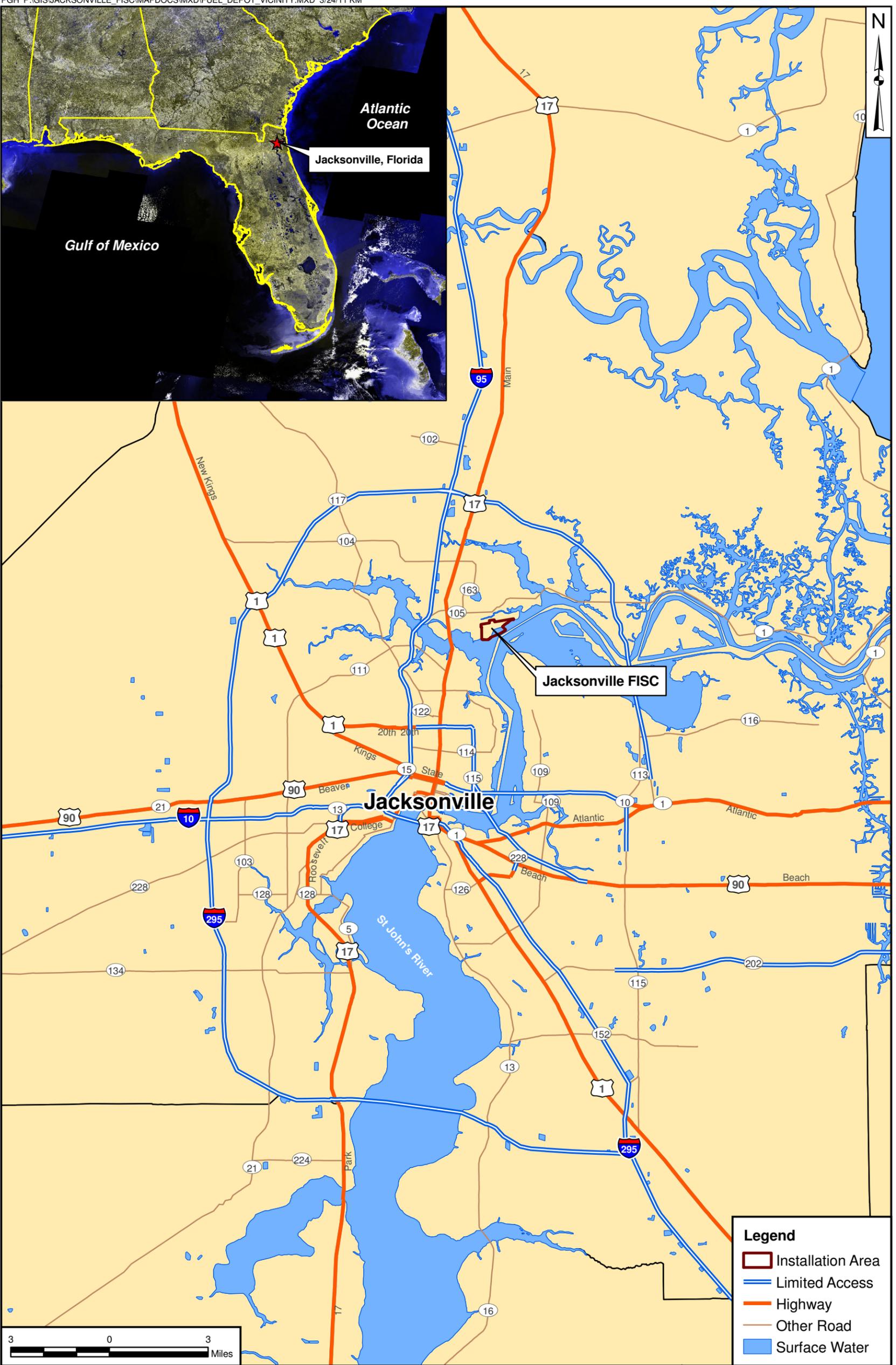
10.4.3 Migration Pathways and Potential Receptors

If contamination is present at Sites 4 and 5, then the following migration routes and release mechanisms have been identified.

Waste materials were directly applied to the surface soils and contamination is potentially present in surface soils. Contaminants could also migrate vertically to subsurface soils through infiltration, and eventually reach groundwater. Once contamination is present in the groundwater, contaminants can migrate to down-gradient areas and even potentially off-site. A fluctuating groundwater table could result in subsurface soil contamination in areas down-gradient from the site. As the groundwater table rises and falls, contaminants may be left behind in the soil above the groundwater table. Contaminants may re-dissolve as the groundwater level rises. Contaminated groundwater could then migrate off-site and impact sediment and surface water.

Potential human receptors include industrial, maintenance, utility, and future construction workers working at the site. Trespassers could have infrequent exposure to media at the site. Trespassing at FISC Navy Fuel Depot is unlikely because of fencing and security measures. Human receptors could be exposed to surface soil (via dermal contact, ingestion, or inhalation), subsurface soil (via vapor intrusion, dermal contact, ingestion, or inhalation), and groundwater (via dermal contact, ingestion, inhalation, or vapor intrusion).

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DRAWN BY T. WHEATON	DATE 02/03/11
CHECKED BY T. DECK	DATE 03/24/11
REVISED BY	DATE
SCALE AS NOTED	



FACILITY LOCATION MAP
 JACKSONVILLE, FISC
 JACKSONVILLE, FLORIDA

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

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LEGEND

- Water Table
- Native Soil
- Site Boundary
- Stressed Vegetation/Visible Sludge
- Native Vegetation

Potential Exposure Pathways and Receptors

<p>Soil Future Site Industrial Worker/Resident</p> <ul style="list-style-type: none"> Inhalation/ingestion of and/or dermal contact with surface and subsurface soils 	<p>Groundwater Future Site Industrial Worker/Resident</p> <ul style="list-style-type: none"> Ingestion and/or dermal contact with groundwater
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Potential Migration Pathways

- Surface Soil → Future Site Industrial Worker/Resident
- Subsurface Soil → Future Site Industrial Worker
- Groundwater → Future Site Industrial Worker/Resident, potential wetlands area, river

DRAWN BY C. Pennington DATE 03-03-11		CONCEPTUAL SITE MODEL SITE 04 AND 05 FISC - NAVY FUEL DEPOT JACKSONVILLE, FLORIDA	CONTRACT NO. CTO JM46
CHECKED BY DATE			OWNER NO.
REVISED BY DATE	SCALE NOT TO SCALE	APPROVED BY DATE	DRAWING NO. Figure 10-3
			REV.

CTO JM46

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

11.1 PROBLEM DEFINITION

The primary objective of this investigation is to determine if contamination is present in surface soil, subsurface soil, and groundwater within the boundaries of Sites 4 and 5 that exceed project action limits (PALs). Compounds that exceed their respective PALs will be retained as COPCs. The secondary objective is to determine the nature and extent of COPCs in accordance with FDEP guidelines. The SAR will document the nature and extent of contamination and will be used to determine if an evaluation of remedial alternatives is required in a Remedial Action Plan (RAP).

11.2 INFORMATION INPUTS

In order to meet the study goals of the investigation, the physical and chemical data to be collected at Sites 4 and 5 are described below:

1. Chemical Analysis: Chemical surface soil, subsurface soil, and groundwater data will be used to determine the nature and extent of VOCs, SVOCs (including PAHs), PCBs, metals, dioxins/furans (surface soil only), TRPH, and speciated TPH (see Worksheet #15). The target analytes represent those analytes that are likely to be associated with historical site operations and as identified in the CSM. The sampling methods that will be utilized are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.
2. Field Parameters: Field investigation parameters for groundwater will include dissolved oxygen, oxidation-reduction potential (ORP), pH, conductivity, temperature, and turbidity. These data will be collected in the field. The relevant Standard Operating Procedures (SOPs) are presented in Worksheet #21.
3. FID Surface and Subsurface Soil Screening: The FID readings will be used to assist in the delineation of the extent of the contamination in surface and subsurface soils. The relevant SOPs are presented in Worksheet #21.

Project Action Limits

Concentrations of target analytes will be compared against PALs. To conduct comparisons of site data to screening values, the selected laboratory must be able to achieve quantitation limits that are low enough to measure constituent concentrations below the PALs. If the laboratory is unable to achieve quantitation limits below screening values the Partnering Team has agreed to replace the PAL with the laboratory limit of quantitation (LOQ) for decision making purposes in accordance with the FDEP protocol (FDEP, 2004).

For this investigation the screening values, which are also known as the PALs, are listed below:

Soil

- SCTLs for Florida Chapter 62-777, F.A.C., Table II (Soils) - Residential direct exposure (FDEP SCTL).
- SCTLs for Florida Chapter 62-777, F.A.C., Table II (Soils) - Leachability based groundwater criteria (FDEP LCH).
- USEPA Regions 3, 6, and 9 (November 2010) Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites - Residential soil values (USEPA-RSL).

When available the FDEP SCTL will be used as the PAL. If a FDEP SCTL is not available the FDEP LCH value will be used. If neither the FDEP SCTL nor FDEP LCH is available the USEPA-RSL will be selected as the PAL.

Groundwater

- Groundwater Cleanup Target Levels (GCTLs) for Florida Chapter 62-777, F.A.C., Table I (FDEP GCTL).
- USEPA Maximum Contaminant Levels (FED MCL).
- USEPA Regions 3, 6, and 9 (November 2010) RSLs for Chemical Contaminants at Superfund Sites - Tapwater (EPA TAP).

When available the FDEP GCTL will be used as the PAL. If a FDEP GCTL is not available the FED MCL value will be used. If neither the FDEP SCTL nor FED MCL is available the EPA TAP will be selected as the PAL.

11.3 STUDY AREA BOUNDARIES

The horizontal boundaries of Sites 4 and 5 are presented in Figure 10-2. Site 4 includes the area in the general vicinity of the former burn kettle, and Site 5 includes the area of the former oil pond. The horizontal boundaries at Sites 4 and 5 as depicted in Figure 10-2 were estimated based on field inspection of surface soils. The final site boundary for Sites 4 and 5 may expand or contract based on the analysis of the soil and groundwater samples. FID field screening results and visual inspection will be used to assist in identifying the horizontal boundaries. Future study boundaries will be identified in a FTMR. The horizontal boundaries are currently limited by the FISC Navy Fuel Depot site boundary as per the scope of this contract.

The vertical boundary for the groundwater investigation is defined by the depth of the water table within the shallow surficial aquifer, which varies, but is typically 3 to 5 feet bgs. Groundwater data will be collected from both the shallow groundwater table (typically less than or equal to 15 feet bgs) and if necessary, deep groundwater data (greater than 15 feet bgs) will be collected as required for delineation. If COPCs exceed PALs in a shallow monitoring well, then deep wells will be installed to vertically delineate any contamination.

The surface soil vertical boundaries are represented by the intervals of interest, which include the 0- to 6-inches bgs interval and the 6-inches to 2-feet bgs interval as required by FDEP 62-780 F.A.C. The subsurface soil vertical boundary to be investigated is soil 2 to 4 feet bgs and 4 to 6 feet bgs (unless the water table [typically 3 to 5 feet bgs] is encountered first). Due to the shallow water table, it is likely that soil samples in the 4 to 6 feet bgs interval will not be collected. Specific subsurface soil intervals will be identified during the investigation when depth to groundwater is determined.

All target analyte concentrations are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future; therefore, no temporal constraints exist. The Site Assessment field activities are anticipated to commence no later than August 2011 and terminate in November 2011.

11.4 ANALYTIC APPROACH

Decision rules are provided below for selecting COPCs and delineating site contamination. In order to determine the appropriate Risk Management Option (RMO) as described in FDEP 62-780, F.A.C. contaminated site Risk Based Corrective Action (RBCA) process, COPCs must be identified and delineated.

COPC Selection Decision Rule

In order to determine if contamination is present in surface soil, subsurface soil, and groundwater as a result of site activities, concentrations will be compared to respective applicable PALs.

- 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 and continue to investigate the site until the COPCs are delineated.

Delineation Decision Rule

The extent of contamination will be determined through comparison of concentrations of COPCs in surface soil, subsurface soil, and groundwater on the outer edge of the sampling pattern to PALs and also by reviewing concentration patterns to assess whether areas of contamination can be enclosed with a concentration isopleth. The isopleth separates the contaminated media from uncontaminated media at the PAL concentration for each COPC.

If the measured concentrations of surface soil, subsurface soil, and groundwater COPCs 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 COPCs, then conduct another phase of field sampling to delineate site contamination for each COPC in each medium.

Risk Based Corrective Action Decision Rule

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. contaminated site RBCA process to determine an appropriate RMO. If a COPC is identified that does not have an appropriate FDEP cleanup target limit the PAL as identified in Worksheet #15 will be used.

Soil

Decision Rule #1

- If the COPCs 95% Upper Confidence Limit (UCL) concentration is less than its residential SCTL and soil concentrations are less than the leachability-based SCTL, then proceed to No Further Action (NFA) without Institutional Controls.
- If the COPCs 95% UCL is greater than its residential SCTL and soil concentrations are greater than its leachability-based SCTL, then proceed to Decision Rule # 2.

Decision Rule #2

- If the COPCs 95% UCL concentration is less than its industrial SCTL, then proceed to NFA with Institutional Controls.
- If the COPCs 95% UCL greater than its industrial SCTL, then the Partnering Team will consider risk to site users and remedial alternatives in a RAP.

Groundwater

- If the maximum measured chemical concentration of a COPC is less than its GCTL, then proceed to NFA without Institutional Controls.
- If the maximum measured chemical concentration of a COPC is greater than its GCTL, then the Partnering Team will consider risk to site users and remedial alternatives in a RAP.

Based on the scope of this investigation, the media being investigated is limited to soils and groundwater; therefore, surface water and sediment will not be evaluated. If contamination is not delineated within the boundary of FISC Navy Fuel Depot, then the Partnering Team will determine if further investigation is warranted under this contract. The soil and groundwater data and selected RMO will be presented in the SAR. Based on the recommendations in the SAR, the Partnering Team will determine if it is necessary to evaluate remedial alternatives in a RAP.

11.5 PERFORMANCE/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 Partnering Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have action levels which are less than the laboratory method detection limits (MDLs), Limits of Detection (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 Partnering 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 Partnering Team members will be involved in rendering the final conclusion regarding adequacy of the data. During future sampling events when determining the extent of COPC contamination in soil, for each interval a minimum of 10 soil samples per one quarter acre will be collected in order to evaluate the soil data according to an acceptable 95% UCL statistical test in accordance with FDEP 62-780, F.A.C. This will insure the soil sampling shall be sufficient to identify the area(s) of highest contaminant concentrations and to allow the calculation of an exposure unit average concentration.

11.6 PLAN FOR OBTAINING DATA

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 Quality Control 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	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	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	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 (≤ 6 °C).	S

1. The Measurement Performance Criteria for laboratory quality control (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)

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Aerial Photographs	Google Earth	Tetra Tech, Geographic Information Systems (GIS) Department, January 2009	Data will be used to determine approximate sample areas and sample locations.	None.

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

The field activities include:

- Mobilization/Demobilization
- Health and Safety Training
- Utility Clearance/Dig Permits
- Electromagnetic Survey
- Monitoring Equipment Calibration
- FID Field Screening and Soil Sampling
- Permanent Well Installation
- Groundwater Sampling
- Investigation-Derived Waste (IDW) Management/Weekly Inspections
- Surveying
- Global Positioning System (GPS) Locating
- Field Decontamination Procedures
- Field Documentation Procedures

Additional project activities include the following tasks:

- Analytical Tasks
- Data Generation Procedures
- Data Management
- Assessment and Oversight
- Data Review
- Project Reports

Mobilization/Demobilization

Multiple mobilizations are required to meet the project objectives. The first mobilization shall consist of clearing trees and underbrush to allow for access to the site. The second mobilization shall consist of the delivery of all equipment, materials, and supplies to the site, the complete assembly in satisfactory working order of all such equipment at the site, and the satisfactory storage at the site of all such materials and supplies. Subsequent mobilizations will be conducted in order to conduct the sampling as described in Worksheet #17. Tetra Tech will coordinate with the facility to identify locations that need to be cleared and for the storage of equipment and supplies.

The demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. 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 to all Tetra Tech field staff and subcontractors will be provided as part of the site mobilization.

Utility Clearance/Dig Permits

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate utility clearance with the Base and Sunshine State One Call. The Base and Utility Companies subscribed to Sunshine State One Call will identify and mark-out utilities that may be present within the soil boring locations. The Tetra Tech FOL will also obtain a dig permit from the Mayport Public Works Department (PWD). See Tetra Tech SOP HS-1.0 (Appendix A) on conducting utility clearance for further information.

Monitoring Equipment Calibration

These procedures are described in Worksheet #22.

FID Field Screening and Soil Sampling

In order to insure all utilities are cleared, surface and subsurface soil samples will be collected using a hand auger to 4 feet. Any subsurface soil being collected at locations where permanent monitoring wells will be installed, the DPT drilling rig used to install the well will be used to collect any interval below 4 feet. Surface and subsurface soils will be collected for site characterization and delineation of COPCs. Each boring will be screened with a FID for VOCs and visual inspections for staining from PAHs as described in more detail in Worksheet #17. After sampling, each borehole not being used for monitoring well installation will be backfilled to within 6 inches of grade using the soil cuttings removed from the borehole. A minimum 6-inch thick grout/bentonite seal will then be placed to grade at each boring. The holes in any paved surface will be backfilled and patched with ready-mix concrete.

Sample jars will be filled using either a decontaminated stainless steel trowel or dedicated disposable plastic trowel. Surface soil will be collected from the 0- to 6-inches bgs interval and the 6-inches to 2-foot bgs interval. Subsurface soil samples will be collected from between 2 to 4 feet bgs and 4 to 6 feet bgs or to the top of the water table, whichever is encountered first. Due to the shallow water table it is likely that the 4 to 6 feet bgs interval will not be collected. The soil borings will be described by the Site Geologist in accordance with Tetra Tech SOP GH-1.5 (Appendix A). A soil boring log will be prepared for each boring

with soil descriptions and all relevant information, observations, depth to saturated soils/water table, and FID field screening results as per SOP SA-6.3 (Appendix A). Sample depths will be included on each log.

All sample locations will be marked with a wooden stake, brightly colored pin flag, or marking paint indicating the sample location. Coordinates will be determined by GPS at each individual sample location, to accurately depict onto site figures and to allow for future repeatable investigations or guide in any remedial action. All sample location markers will be removed prior to the final demobilization.

All of the soil samples will be collected using the procedures specified in FDEP SOP FS 3000 for soil sampling (Appendix A). Worksheets #17 and #18 specify the soil sampling locations and analyte groups for this investigation. Worksheet #19 specifies the analytical methods to be used.

Permanent Well Installation

Multiple 1.5 inch diameter permanent monitoring wells will be installed using DPT at Sites 4 and 5 during this investigation. All shallow wells will be installed via SOP GH-2.8 (Groundwater Monitoring Well Installation) (Appendix A) with 10-foot screens intersecting the surficial water table to a depth of approximately 15 feet bgs. As necessary, all deep wells will be 1.5 inch diameter installed using DPT to a depth to be determined based on results of the groundwater investigation.

Groundwater Sampling

Groundwater samples will be collected from permanent monitoring wells utilizing a peristaltic pump and sterile polyethylene and medical-grade silicon tubing. In general, groundwater samples will be collected from the 5- to 10-foot bgs interval. The actual sampling depth at each monitoring well location is subject to change based on the depth to groundwater measured in each well. All of the groundwater samples will be collected using the procedures specified in FDEP SOP FS 2200 for groundwater sampling (Appendix A). Worksheets #17 and #18 specify the groundwater sampling locations and analyte groups for this investigation. Worksheet #19 specifies the analytical methods to be used.

Investigation-Derived Waste Management

Types of IDW generated during this investigation that could be potentially contaminated include: groundwater purged from monitoring wells, excess soil cuttings not collected in the laboratory supplied sample containers, one-time use sampling supplies, decontamination wastewaters, and personal protective equipment (PPE) and clothing. Groundwater removed and not sampled during this investigation and excess soil will be placed into labeled, sealable 55-gallon Department of Transportation approved steel drums provided by the PWD at Mayport. The drums will be inspected weekly until picked

up and 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 profiling results of the groundwater and soil waste is received from the laboratory and accepted. PPE and clothing will be wiped clean and disposed of in trash containers as general refuse.

Surveying

The locations and elevations of all monitoring wells will be surveyed. The North American Datum (NAD) 1983 will be used as the horizontal datum. Sample locations will be surveyed horizontally to the nearest 0.10 foot. Vertical elevations will be referenced to 1988 National Geodetic Vertical Datum (NGVD) to an accuracy of 0.01 foot.

Global Position System Locating

A GPS unit will be used to locate all soil sampling points not associated with surveyed monitoring well locations. The GPS equipment will be checked on control monuments before and after each days use, and these checks will be documented in the field notebook per SOP GPS-1 (Appendix A). To ensure sub-meter accuracy, a minimum of six satellites are required to capture a position.

Field Decontamination Procedure

Decontamination of major equipment and sampling equipment will be in general accordance with FDEP SOP FC 1000 and Tetra Tech SOP SA-7.1 (Appendix A).

Field Documentation Procedures

Pre-preserved, certified-clean bottle ware will be supplied by Empirical, Katahdin, CFA, and Enco. Matrix-specific sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded in bound field notebooks or specific field forms. Samples will be packaged and shipped according to FDEP SOP FS 1000 (Appendix A).

Field documentation will also be performed in accordance with FDEP SOP FD 1000 and Tetra Tech SOP SA-6.3 (Appendix A). A summary of all field activities will be properly recorded in indelible ink in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

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
- Project start and end dates
- Names and responsibilities of on-site project personnel including subcontractor personnel
- Arrival/departure of site visitors' chronology
- Arrival/departure of equipment chronology
- Sampling activities chronology, weather conditions, and sample log sheet references
- Description of subcontractor activities chronology
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date
- Description of borehole or monitoring well installation activities and operations
- Health and safety issues
- Description of photographs including date, time, photographer, roll and picture number, location, and compass direction of photograph

All entries will be written in indelible ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change.

Analytic Tasks

Chemical analyses will be performed by Empirical, except for the following: dioxin/furan analyses will be performed by CFA; TPH speciation, if deemed necessary based on the TRPH analysis results determined by Empirical, will be performed by Katahdin; and Enco will perform low level 1,4-dioxane analysis of groundwater samples by SW-846 Method 8270C Selected Ion Monitoring (SIM). Empirical, CFA, and Katahdin are all Department of Defense (DoD) Environmental Laboratory Program (ELAP) and Florida Department of Health (FDOH) accredited laboratories. Enco is a FDOH accredited laboratory. Copies of the laboratory accreditations are located in Appendix B. Analyses will be performed in accordance with the analytical methods specified in Worksheet #19. The laboratories will meet the PALs as shown in Worksheet #15. The laboratories will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheet #19 and #30. Copies of the non-proprietary laboratories SOPs are included in Appendix B. All proprietary laboratory

SOPs (all SOPs that are not in Appendix B, but are identified in Worksheet #23) have been reviewed by the Tetra Tech Project Chemist and were found to be suitable for this project.

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 which will eventually be uploaded to Naval Installation Restoration Information Solution (NIRIS). Percent moisture information will also be captured in the SAR.

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 quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.).

Data Management

Data management tasks, including the data handling, tracking, storage, archiving, retrieval, and security processes, are addressed in Worksheet # 29.

Data Assessment and Oversight

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

Data Review

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

Project Reports

A SAR will be prepared to document the results of this sampling event. The report will include sections summarizing Site Assessment (SA) activities, physical characteristics, nature and extent of contamination, and conclusions and recommendations. Based on the recommendations in the SAR, the Partnering Team will determine if it is necessary to evaluate remedial alternatives in a RAP.

Each report will be issued in draft to NAVFAC SE for initial review. NAVFAC SE comments will be addressed, and the draft final report will be issued to the FDEP for regulatory review.

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

Matrix: Groundwater

Analytical: VOCs (including low level 1,4-Dioxane) (1)

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/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	210,000	FDEP GCTL	70,000	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	0.5
1,2-DIBROMOETHANE	106-93-4	0.02	FDEP GCTL	0.0067	1	0.5	0.25
1,2-DICHLOROBENZENE	95-50-1	600	FDEP GCTL	200	1	0.5	0.25
1,2-DICHLOROETHANE	107-06-2	3	FDEP GCTL	1.0	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 (1)	123-91-1	3.2	FDEP GCTL	1.1	3	2	1
2-BUTANONE	78-93-3	4,200	FDEP GCTL	1,400	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	6,300	FDEP GCTL	2,100	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	0.20	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	1.0	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	4.0	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	0.90	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP GCTL	23	1	0.5	0.25

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
CIS-1,3-DICHLOROPROPENE	10061-01-5	NC	None	NC	1	0.5	0.25
CYCLOHEXANE	110-82-7	1,300	Tap Water RSL	430	1	0.5	0.25
DICHLORODIFLUOROMETHANE	75-71-8	1,400	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		10,000	FED MCL	3,300	2	1	0.5
METHYL ACETATE	79-20-9	3,000	FDEP GCTL	1,000	2	1	0.5
METHYL CYCLOHEXANE	108-87-2	NC	None	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	10,000	FED MCL	3,300	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	1.0	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	1.0	1	0.5	0.25
TRICHLOROFLUOROMETHANE	75-69-4	2,100	FDEP GCTL	700	2	1	0.5
VINYL CHLORIDE	75-01-4	1	FDEP GCTL	0.33	1	0.5	0.25

CAS = Chemical Abstracts Service

PQLG = Project Quantitation Limit Goal

NC = No Criteria

1) Low level 1,4-dioxane analysis will be performed by Enco following SW-846 Method 8270C SIM.

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, Maximum Contaminant Level (MCL) (USEPA, 2011); and Tap Water RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Tapwater (USEPA, November 2010).

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

Shaded and Bold row indicate the PAL is less than the MDL; therefore, 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, October 2004).

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

Matrix: Groundwater
Analytical: SVOCs (including PAHs)

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/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	0.70	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	0.5
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	0.10	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	7.0	20	10	5
2-NITROPHENOL	88-75-5	NC	None	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	5	2.5	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	None	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	None	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	5	2.5	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	1.0	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	0.010	5	2.5	1.25
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	6	FDEP GCTL	2.0	5	2.5	1.25
BUTYL BENZYL PHTHALATE	85-68-7	140	FDEP GCTL	47	5	2.5	1.25
CAPROLACTAM	105-60-2	1,800	Tap Water RSL	600	5	2.5	1.25
CARBAZOLE	86-74-8	1.8	FDEP GCTL	0.60	5	2.5	1.25
DIBENZOFURAN	132-64-9	28	FDEP GCTL	9.3	5	2.5	1.25
DIETHYL PHTHALATE	84-66-2	5,600	FDEP GCTL	1,900	5	2.5	1.25

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
DIMETHYL PHTHALATE	131-11-3	70,000	FDEP GCTL	23,000	5	2.5	1.25
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
NAPHTHALENE	91-20-3	14	FDEP GCTL	4.7	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	4	2	1
PHENOL	108-95-2	10	FDEP GCTL	3.3	5	2.5	1.25
PAHs							
1-METHYLNAPHTHALENE	90-12-0	28	FDEP GCTL	9.3	5	2.5	1.25
2-METHYLNAPHTHALENE	91-57-6	28	FDEP GCTL	9.3	5	2.5	1.25
ACENAPHTHENE	83-32-9	20	FDEP GCTL	6.7	5	2.5	1.25
ACENAPHTHYLENE	208-96-8	210	FDEP GCTL	70	5	2.5	1.25
ANTHRACENE	120-12-7	2,100	FDEP GCTL	700	5	2.5	1.25
BENZO(A)ANTHRACENE (1)	56-55-3	0.05	FDEP GCTL	0.017	0.2	0.1	0.05
BENZO(A)PYRENE (1)	50-32-8	0.2	FDEP GCTL	0.067	0.2	0.1	0.05
BENZO(B)FLUORANTHENE (1)	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	5	2.5	1.25
BENZO(K)FLUORANTHENE (1)	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	5	2.5	1.25
DIBENZO(A,H)ANTHRACENE (1)	53-70-3	0.005	FDEP GCTL	0.0017	0.1	0.05	0.005
FLUORANTHENE	206-44-0	280	FDEP GCTL	93	5	2.5	1.25
FLUORENE	86-73-7	280	FDEP GCTL	93	5	2.5	1.25
INDENO(1,2,3-CD)PYRENE (1)	193-39-5	0.05	FDEP GCTL	0.017	0.2	0.1	0.05
PHENANTHRENE	85-01-8	210	FDEP GCTL	70	5	2.5	1.25
PYRENE	129-00-0	210	FDEP GCTL	70	5	2.5	1.25
NAPHTHALENE	91-20-3	14	FDEP GCTL	4.7	5	2.5	1.25

(1) 8270D Low Level Full Scan SOP will be utilized for certain PAHs.

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, November 2010).

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

Shaded and Bold row indicate the PAL is less than the MDL; therefore, 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, October 2004).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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 (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
AROCLOR-1016	12674-11-2	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1221	11104-28-2	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1232	11141-16-5	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1242	53469-21-9	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1248	12672-29-6	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1254	11097-69-1	0.5	FED MCL	0.17	0.5	0.25	0.125
AROCLOR-1260	11096-82-5	0.5	FED MCL	0.17	0.5	0.25	0.125
TOTAL AROCLORS	1336-36-3	0.5	FED MCL	0.17	NA	NA	NA

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, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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: Metals

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
ANTIMONY*	7440-36-0	6	FDEP GCTL	2.0	2.5	2	1.25
ARSENIC	7440-38-2	10	FDEP GCTL	3.3	10	6	3
BARIUM	7440-39-3	2,000	FDEP GCTL	670	40	10	5
BERYLLIUM	7440-41-7	4	FDEP GCTL	1.3	5	2	1
CADMIUM	7440-43-9	5	FDEP GCTL	1.7	5	2	1
CHROMIUM	7440-47-3	100	FDEP GCTL	33	10	4	2
COPPER	7440-50-8	1,000	FDEP GCTL	330	10	8	4
LEAD	7439-92-1	15	FDEP GCTL	5.0	3	3	1.5
MERCURY	7439-97-6	2	FDEP GCTL	0.67	0.2	0.2	0.08
NICKEL	7440-02-0	100	FDEP GCTL	33	10	6	3
SELENIUM	7782-49-2	50	FDEP GCTL	17	10	5	3
SILVER	7440-22-4	100	FDEP GCTL	33	10	2	1
THALLIUM*	7440-28-0	2	FDEP GCTL	0.67	2	1	0.75
TIN	7440-31-5	4,200	FDEP GCTL	1,400	7	5	2.5
ZINC	7440-66-6	5,000	FDEP GCTL	1,700	20	10	5

*Antimony and Thallium will be concentrated 4 times

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, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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

Analyte	CAS Number	PAL (ug/L)	PAL Reference	PQLGs (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
TOTAL PETROLEUM HYDROCARBONS		5000	FDEP GCTL	1670	680	340	170

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, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
1,1,1-TRICHLOROETHANE	71-55-6	730	FDEP SCTL	240	0.333	0.167	0.0833
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.7	FDEP SCTL	0.23	0.333	0.167	0.0833
1,1,2-TRICHLOROETHANE	79-00-5	1.4	FDEP SCTL	0.47	0.333	0.167	0.0833
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	18,000	FDEP SCTL	6,000	0.333	0.167	0.0833
1,1-DICHLOROETHANE	75-34-3	390	FDEP SCTL	130	0.333	0.167	0.0833
1,1-DICHLOROETHENE	75-35-4	95	FDEP SCTL	32	0.333	0.167	0.0833
1,2,3-TRICHLOROBENZENE	87-61-6	650	FDEP SCTL	220	0.333	0.167	0.0833
1,2,4-TRICHLOROBENZENE	120-82-1	660	FDEP SCTL	220	0.333	0.167	0.0833
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	0.7	FDEP SCTL	0.23	0.333	0.167	0.0833
1,2-DIBROMOETHANE	106-93-4	0.1	FDEP SCTL	0.033	0.333	0.167	0.0833
1,2-DICHLOROBENZENE	95-50-1	880	FDEP SCTL	290	0.333	0.167	0.0833
1,2-DICHLOROETHANE	107-06-2	0.5	FDEP SCTL	0.17	0.333	0.167	0.0833
1,2-DICHLOROPROPANE	78-87-5	0.6	FDEP SCTL	0.20	0.333	0.167	0.0833
1,3-DICHLOROBENZENE	541-73-1	380	FDEP SCTL	130	0.333	0.167	0.0833
1,4-DICHLOROBENZENE	106-46-7	6.4	FDEP SCTL	2.2	0.333	0.167	0.0833
1,4-DIOXANE	123-91-1	23	FDEP SCTL	7.7	1	0.5	0.25
2-BUTANONE	78-93-3	16,000	FDEP SCTL	5,300	0.333	0.167	0.0833
2-HEXANONE	591-78-6	24	FDEP SCTL	8.0	0.333	0.167	0.0833
4-METHYL-2-PENTANONE	108-10-1	4,300	FDEP SCTL	1,400	1.33	0.667	0.333
ACETONE	67-64-1	11,000	FDEP SCTL	3,700	0.333	0.167	0.0833
BENZENE	71-43-2	1.2	FDEP SCTL	0.40	0.333	0.167	0.0833
BROMOCHLOROMETHANE	74-97-5	95	FDEP SCTL	32	0.005	0.0025	0.00125
BROMODICHLOROMETHANE	75-27-4	1.5	FDEP SCTL	0.50	0.005	0.0025	0.00125
BROMOFORM	75-25-2	48	FDEP SCTL	16	0.01	0.005	0.0025
BROMOMETHANE	74-83-9	3.1	FDEP SCTL	1.0	0.005	0.0025	0.00125
CARBON DISULFIDE	75-15-0	270	FDEP SCTL	90	0.005	0.0025	0.00125
CARBON TETRACHLORIDE	56-23-5	0.5	FDEP SCTL	0.17	0.005	0.0025	0.00125
CHLOROBENZENE	108-90-7	120	FDEP SCTL	40	0.005	0.0025	0.00125
CHLORODIBROMOMETHANE	124-48-1	1.5	FDEP SCTL	0.50	0.005	0.0025	0.00125
CHLOROETHANE	75-00-3	3.9	FDEP SCTL	1.3	0.005	0.0025	0.00125
CHLOROFORM	67-66-3	0.4	FDEP SCTL	0.13	0.005	0.0025	0.00125
CHLOROMETHANE	74-87-3	4	FDEP SCTL	1.3	0.005	0.0025	0.00125
CIS-1,2-DICHLOROETHENE	156-59-2	33	FDEP SCTL	11	0.005	0.0025	0.00125
CIS-1,3-DICHLOROPROPENE	10061-01-5	1.7	EPA RSL	0.57	0.01	0.005	0.0025
CYCLOHEXANE	110-82-7	700	EPA RSL	230	0.005	0.0025	0.00125
DICHLORODIFLUOROMETHANE	75-71-8	77	FDEP SCTL	26	0.005	0.0025	0.00125
ETHYLBENZENE	100-41-4	1,500	FDEP SCTL	500	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 MDL (mg/kg)
ISOPROPYLBENZENE	98-82-8	220	FDEP SCTL	73	0.005	0.0025	0.00125
M+P-XYLENES		NC	None	NC	0.005	0.0025	0.00125
METHYL ACETATE	79-20-9	6,800	FDEP SCTL	2,300	0.01	0.005	0.0025
METHYL CYCLOHEXANE	108-87-2	NC	None	NC	0.005	0.0025	0.00125
METHYL TERT-BUTYL ETHER	1634-04-4	4,400	FDEP SCTL	1,500	0.005	0.0025	0.00125
METHYLENE CHLORIDE	75-09-2	17	FDEP SCTL	5.7	0.005	0.0025	0.00125
O-XYLENE	95-47-6	380	EPA RSL	130	0.005	0.0025	0.00125
STYRENE	100-42-5	3,600	FDEP SCTL	1,200	0.005	0.0025	0.00125
TETRACHLOROETHENE	127-18-4	8.8	FDEP SCTL	2.9	0.005	0.0025	0.00125
TOLUENE	108-88-3	7,500	FDEP SCTL	2,500	0.01	0.005	0.0025
TRANS-1,2-DICHLOROETHENE	156-60-5	53	FDEP SCTL	18	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	6.4	FDEP SCTL	2.1	0.005	0.0025	0.00125
TRICHLOROFLUOROMETHANE	75-69-4	270	FDEP SCTL	90	0.01	0.005	0.0025
VINYL CHLORIDE	75-01-4	0.2	FDEP SCTL	0.067	0.01	0.005	0.0025

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

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

Shaded and Bold row indicate the PAL is less than the MDL; therefore, 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, October 2004).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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 PAHs)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
1,1-BIPHENYL	92-52-4	3,000	FDEP SCTL	1,000	0.333	0.167	0.0833
1,2,4,5-TETRACHLOROBENZENE	95-94-3	12	FDEP SCTL	4.0	0.333	0.167	0.0833
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	6	FDEP SCTL	2.0	0.333	0.167	0.0833
2,3,4,6-TETRACHLOROPHENOL	58-90-2	2,100	FDEP SCTL	700	0.333	0.167	0.0833
2,4,5-TRICHLOROPHENOL	95-95-4	7,700	FDEP SCTL	2,600	0.333	0.167	0.0833
2,4,6-TRICHLOROPHENOL	88-06-2	70	FDEP SCTL	23	0.333	0.167	0.0833
2,4-DICHLOROPHENOL	120-83-2	190	FDEP SCTL	63	0.333	0.167	0.0833
2,4-DIMETHYLPHENOL	105-67-9	1,300	FDEP SCTL	430	1.33	0.667	0.333
2,4-DINITROPHENOL	51-28-5	110	FDEP SCTL	37	3.33	1.67	0.833
2,4-DINITROTOLUENE	121-14-2	1.2	FDEP SCTL	0.40	0.333	0.167	0.0833
2,6-DINITROTOLUENE	606-20-2	1.2	FDEP SCTL	0.40	0.333	0.167	0.0833
2-CHLORONAPHTHALENE	91-58-7	5,000	FDEP SCTL	1,700	0.333	0.167	0.0833
2-CHLOROPHENOL	95-57-8	130	FDEP SCTL	43	0.333	0.167	0.0833
2-METHYLPHENOL	95-48-7	2,900	FDEP SCTL	970	0.333	0.167	0.0833
2-NITROANILINE	88-74-4	24	FDEP SCTL	8.0	1.33	0.667	0.333
2-NITROPHENOL	88-75-5	NC	None	NC	0.333	0.167	0.0833
3,3'-DICHLOROBENZIDINE	91-94-1	2.1	FDEP SCTL	0.70	0.333	0.167	0.0833
3-NITROANILINE	99-09-2	21	FDEP SCTL	7.0	1.33	0.667	0.333
4,6-DINITRO-2-METHYLPHENOL	534-52-1	8.4	FDEP SCTL	2.8	3.33	1.67	0.833
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	None	NC	0.333	0.167	0.0833
4-CHLORO-3-METHYLPHENOL	59-50-7	600	FDEP SCTL	200	0.333	0.167	0.0833
4-CHLOROANILINE	106-47-8	270	FDEP SCTL	90	0.333	0.167	0.0833
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	None	NC	0.333	0.167	0.0833
4-METHYLPHENOL	106-44-5	300	FDEP SCTL	100	0.333	0.167	0.0833
4-NITROANILINE	100-01-6	17	FDEP SCTL	5.7	1.33	0.667	0.333
4-NITROPHENOL	100-02-7	560	FDEP SCTL	190	1.33	0.667	0.333
ACETOPHENONE	98-86-2	3,900	FDEP SCTL	1,300	0.333	0.167	0.0833
ATRAZINE	1912-24-9	4.3	FDEP SCTL	1.4	0.333	0.167	0.0833
BENZALDEHYDE	100-52-7	3300	FDEP SCTL	1,100	0.333	0.167	0.0833
BIS(2-CHLOROETHOXY)METHANE	111-91-1	250	FDEP SCTL	83	0.333	0.167	0.0833
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.3	FDEP SCTL	0.10	0.333	0.167	0.0833
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	72	FDEP SCTL	24	0.333	0.167	0.0833
BUTYL BENZYL PHTHALATE	85-68-7	17,000	FDEP SCTL	5,700	0.333	0.167	0.0833
CAPROLACTAM	105-60-2	3,100	EPA RSL	1,000	0.333	0.167	0.0833
CARBAZOLE	86-74-8	49	FDEP SCTL	16	0.333	0.167	0.0833
DIBENZOFURAN	132-64-9	320	FDEP SCTL	110	0.333	0.167	0.0833
DIETHYL PHTHALATE	84-66-2	61,000	FDEP SCTL	20,000	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 MDL (mg/kg)
DIMETHYL PHTHALATE	131-11-3	690,000	FDEP SCTL	230,000	0.333	0.167	0.0833
DI-N-BUTYL PHTHALATE	84-74-2	8,200	FDEP SCTL	2,700	0.333	0.167	0.0833
DI-N-OCTYL PHTHALATE	117-84-0	1,700	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	6.2	FDEP SCTL	2.1	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	38	FDEP SCTL	13	0.333	0.167	0.0833
ISOPHORONE	78-59-1	540	FDEP SCTL	180	0.333	0.167	0.0833
NAPHTHALENE	91-20-3	55	FDEP SCTL	18	0.333	0.167	0.0833
NITROBENZENE	98-95-3	18	FDEP SCTL	6.0	0.333	0.167	0.0833
N-NITROSODIPHENYLAMINE	86-30-6	180	FDEP SCTL	60	0.333	0.167	0.0833
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.08	FDEP SCTL	0.027	0.333	0.167	0.067
PENTACHLOROPHENOL	87-86-5	7.2	FDEP SCTL	2.4	1.33	0.667	0.333
PHENOL	108-95-2	500	FDEP SCTL	170	0.333	0.167	0.0833
PAHs							
1-METHYLNAPHTHALENE	90-12-0	200	FDEP SCTL	67	0.333	0.167	0.0833
2-METHYLNAPHTHALENE	91-57-6	210	FDEP SCTL	70	0.333	0.167	0.0833
ACENAPHTHENE	83-32-9	2,400	FDEP SCTL	800	0.333	0.167	0.0833
ACENAPHTHYLENE	208-96-8	1,800	FDEP SCTL	600	0.333	0.167	0.0833
ANTHRACENE	120-12-7	21,000	FDEP SCTL	7,000	0.333	0.167	0.0833
BENZO(A)ANTHRACENE	56-55-3	0.15	EPA RSL	0.050	0.333	0.167	0.0833
BENZO(A)PYRENE	50-32-8	0.1	FDEP SCTL	0.033	0.333	0.167	0.0833
BENZO(B)FLUORANTHENE	205-99-2	0.15	EPA RSL	0.050	0.333	0.167	0.0833
BENZO(G,H,I)PERYLENE	191-24-2	2,500	FDEP SCTL	830	0.333	0.167	0.0833
BENZO(K)FLUORANTHENE	207-08-9	1.5	EPA RSL	0.50	0.333	0.167	0.0833
CHRYSENE	218-01-9	15	EPA RSL	5.0	0.333	0.167	0.0833
DIBENZO(A,H)ANTHRACENE	53-70-3	0.015	EPA RSL	0.0050	0.167	0.0833	0.015
FLUORANTHENE	206-44-0	3,200	FDEP SCTL	1,100	0.333	0.167	0.0833
FLUORENE	86-73-7	2,600	FDEP SCTL	870	0.333	0.167	0.0833
INDENO(1,2,3-CD)PYRENE	193-39-5	0.15	EPA RSL	0.050	0.333	0.167	0.0833
PHENANTHRENE	85-01-8	2,200	FDEP SCTL	730	0.333	0.167	0.0833
PYRENE	129-00-0	2,400	FDEP SCTL	800	0.333	0.167	0.0833
NAPHTHALENE	91-20-3	55	FDEP SCTL	18	0.333	0.167	0.0833

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

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

Shaded and Bold row indicate the PAL is less than the MDL; therefore, 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, October 2004).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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 (High in Petroleum Hydrocarbons)
Analytical: SVOCs (including PAHs)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
1,1-BIPHENYL	92-52-4	3,000	FDEP SCTL	1,000	20	10	5
1,2,4,5-TETRACHLOROBENZENE	95-94-3	12	FDEP SCTL	4.0	20	10	5
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	6	FDEP SCTL	2.0	20	10	5
2,3,4,6-TETRACHLOROPHENOL	58-90-2	2,100	FDEP SCTL	700	20	10	5
2,4,5-TRICHLOROPHENOL	95-95-4	7,700	FDEP SCTL	2,600	20	10	5
2,4,6-TRICHLOROPHENOL	88-06-2	70	FDEP SCTL	23	20	10	5
2,4-DICHLOROPHENOL	120-83-2	190	FDEP SCTL	63	20	10	5
2,4-DIMETHYLPHENOL	105-67-9	1,300	FDEP SCTL	430	80	40	20
2,4-DINITROPHENOL	51-28-5	110	FDEP SCTL	37	200	100	50
2,4-DINITROTOLUENE	121-14-2	1.2	FDEP SCTL	0.40	20	10	5
2,6-DINITROTOLUENE	606-20-2	1.2	FDEP SCTL	0.40	20	10	5
2-CHLORONAPHTHALENE	91-58-7	5,000	FDEP SCTL	1,700	20	10	5
2-CHLOROPHENOL	95-57-8	130	FDEP SCTL	43	20	10	5
2-METHYLPHENOL	95-48-7	2,900	FDEP SCTL	970	20	10	5
2-NITROANILINE	88-74-4	24	FDEP SCTL	8.0	80	40	20
2-NITROPHENOL	88-75-5	NC	None	NC	20	10	5
3,3'-DICHLOROBENZIDINE	91-94-1	2.1	FDEP SCTL	0.70	20	10	5
3-NITROANILINE	99-09-2	21	FDEP SCTL	7.0	80	40	20
4,6-DINITRO-2-METHYLPHENOL	534-52-1	8.4	FDEP SCTL	2.8	200	100	50
4-BROMOPHENYL PHENYL ETHER	101-55-3	NC	None	NC	20	10	5
4-CHLORO-3-METHYLPHENOL	59-50-7	600	FDEP SCTL	200	20	10	5
4-CHLOROANILINE	106-47-8	270	FDEP SCTL	90	20	10	5
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NC	None	NC	20	10	5
4-METHYLPHENOL	106-44-5	300	FDEP SCTL	100	20	10	5
4-NITROANILINE	100-01-6	17	FDEP SCTL	5.7	80	40	20
4-NITROPHENOL	100-02-7	560	FDEP SCTL	190	80	40	20
ACETOPHENONE	98-86-2	3,900	FDEP SCTL	1,300	20	10	5
ATRAZINE	1912-24-9	4.3	FDEP SCTL	1.4	20	10	5
BENZALDEHYDE	100-52-7	3,300	FDEP SCTL	1,100	20	10	5
BIS(2-CHLOROETHOXY)METHANE	111-91-1	250	FDEP SCTL	83	20	10	5
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.3	FDEP SCTL	0.10	20	10	5
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	72	FDEP SCTL	24	20	10	5
BUTYL BENZYL PHTHALATE	85-68-7	17,000	FDEP SCTL	5,700	20	10	5
CAPROLACTAM	105-60-2	3,100	EPA RSL	1,000	20	10	5
CARBAZOLE	86-74-8	49	FDEP SCTL	16	20	10	5
DIBENZOFURAN	132-64-9	320	FDEP SCTL	110	20	10	5
DIETHYL PHTHALATE	84-66-2	61,000	FDEP SCTL	20,000	20	10	5

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
DIMETHYL PHTHALATE	131-11-3	690,000	FDEP SCTL	230,000	20	10	5
DI-N-BUTYL PHTHALATE	84-74-2	8,200	FDEP SCTL	2,700	20	10	5
DI-N-OCTYL PHTHALATE	117-84-0	1,700	FDEP SCTL	570	20	10	5
HEXACHLOROBENZENE	118-74-1	0.4	FDEP SCTL	0.13	20	10	5
HEXACHLOROBUTADIENE	87-68-3	6.2	FDEP SCTL	2.1	20	10	5
HEXACHLOROCYCLOPENTADIENE	77-47-4	9.5	FDEP SCTL	3.2	20	10	5
HEXACHLOROETHANE	67-72-1	38	FDEP SCTL	13	20	10	5
ISOPHORONE	78-59-1	540	FDEP SCTL	180	20	10	5
NAPHTHALENE	91-20-3	55	FDEP SCTL	18	20	10	5
NITROBENZENE	98-95-3	18	FDEP SCTL	6.0	20	10	5
N-NITROSODIPHENYLAMINE	86-30-6	180	FDEP SCTL	60	20	10	5
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.08	FDEP SCTL	0.027	20	10	4
PENTACHLOROPHENOL	87-86-5	7.2	FDEP SCTL	2.4	80	40	20
PHENOL	108-95-2	500	FDEP SCTL	170	20	10	5
PAHs							
1-METHYLNAPHTHALENE	90-12-0	200	FDEP SCTL	67	20	10	5
2-METHYLNAPHTHALENE	91-57-6	210	FDEP SCTL	70	20	10	5
ACENAPHTHENE	83-32-9	2,400	FDEP SCTL	800	20	10	5
ACENAPHTHYLENE	208-96-8	1,800	FDEP SCTL	600	20	10	5
ANTHRACENE	120-12-7	21,000	FDEP SCTL	7,000	20	10	5
BENZO(A)ANTHRACENE	56-55-3	0.15	EPA RSL	0.050	20	10	5
BENZO(A)PYRENE	50-32-8	0.1	FDEP SCTL	0.033	20	10	5
BENZO(B)FLUORANTHENE	205-99-2	0.15	EPA RSL	0.050	20	10	5
BENZO(G,H,I)PERYLENE	191-24-2	2,500	FDEP SCTL	830	20	10	5
BENZO(K)FLUORANTHENE	207-08-9	1.5	EPA RSL	0.50	20	10	5
CHRYSENE	218-01-9	15	EPA RSL	5.0	20	10	5
DIBENZO(A,H)ANTHRACENE	53-70-3	0.015	EPA RSL	0.0050	20	10	5
FLUORANTHENE	206-44-0	3,200	FDEP SCTL	1,100	20	10	5
FLUORENE	86-73-7	2,600	FDEP SCTL	870	20	10	5
INDENO(1,2,3-CD)PYRENE	193-39-5	0.15	EPA RSL	0.050	20	10	5
PHENANTHRENE	85-01-8	2,200	FDEP SCTL	730	20	10	5
PYRENE	129-00-0	2,400	FDEP SCTL	800	20	10	5
NAPHTHALENE	91-20-3	55	FDEP SCTL	18	20	10	5

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

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

Shaded and Bold row indicate the PAL is less than the MDL; therefore, 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, October 2004).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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.

* = All samples submitted/received will be visually evaluated by field/lab personnel to determine if sample would be expected to have normal or high TRPH concentration. If high TRPH concentration is expected, the sample will be extracted for Florida Residual Petroleum Organic Method (FL-PRO) method using a 5g-20 mL microtip sonication. Resulting concentrations will be used to determine extraction method for SVOCs. If TRPH exceeds 10,000 mg/kg, 5g-20 mL microtip sonication will be used for SVOC extraction/analysis. If TRPH does not exceed 10,000 mg/kg, standard microwave extraction will be used. In cases where TRPH concentration is below the LOQ of 1,360 mg/kg, the 20 milliliter (mL) extract will be concentrated 5-fold to provide results with a nominal LOQ of 272 mg/kg – below the PAL of 340 mg/kg.

Matrix: Soil
Analytical: PCBs

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (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
TOTAL AROCLORS	1336-36-3	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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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: Metals

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
ANTIMONY*	7440-36-0	27	FDEP SCTL	9.0	2	1.6	1
ARSENIC	7440-38-2	2.1	FDEP SCTL	0.70	2	1.2	0.6
BARIUM	7440-39-3	120	FDEP SCTL	40	8	4	1
BERYLLIUM	7440-41-7	120	FDEP SCTL	40	1	0.4	0.2
CADMIUM	7440-43-9	82	FDEP SCTL	27	1	0.4	0.2
CHROMIUM	7440-47-3	210	FDEP SCTL	70	2	0.8	0.4
COPPER	7440-50-8	150	FDEP SCTL	50	2	1.6	0.8
LEAD	7439-92-1	400	FDEP SCTL	130	1	0.6	0.3
MERCURY	7439-97-6	3	FDEP SCTL	1.0	0.033	0.033	0.013
NICKEL	7440-02-0	340	FDEP SCTL	110	2	1.2	0.6
SELENIUM	7782-49-2	440	FDEP SCTL	150	2	1	0.6
SILVER	7440-22-4	410	FDEP SCTL	140	2	0.4	0.2
THALLIUM*	7440-28-0	6.1	FDEP SCTL	2.0	1.6	0.8	0.6
TIN	7440-31-5	47,000	FDEP SCTL	16,000	40	10	2
ZINC	7440-66-6	26,000	FDEP SCTL	8,700	4	2	1

*Antimony and Thallium will be concentrated 4 times

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
TOTAL PETROLEUM HYDROCARBONS	TTNUS001	340	FDEP SCTL	110	45.3	22.7	11.3

Matrix: Soil (High in Petroleum Hydrocarbons)
Analytical: TRPH

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Empirical LOQ (mg/kg)	Empirical LOD (mg/kg)	Empirical MDL (mg/kg)
TOTAL PETROLEUM HYDROCARBONS	TTNUS001	340	FDEP SCTL	110	1,360	680	340

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

* = All samples submitted/received will be visually evaluated by field/lab personnel to determine if sample would be expected to have normal or high TRPH concentration. If high TRPH concentration is expected, the sample will be extracted for FL-PRO method using a 5g-20 mL microtip sonication. Resulting concentrations will be used to determine extraction method for SVOCs. If TRPH exceeds 10,000 mg/kg, 5g-20 mL microtip sonication will be used for SVOC extraction/analysis. If TRPH does not exceed 10,000 mg/kg, standard microwave extraction will be used. In cases where TRPH concentration is below the LOQ of 1,360 mg/kg, the 20 mL extract will be concentrated 5-fold to provide results with a nominal LOQ of 272 mg/kg – below the PAL of 340 mg/kg.

Matrix: Soil
Analytical: Speciated TPH (VPH and EPH -by MADEP Method)

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	Katahdin LOQ (mg/kg)	Katahdin LOD (mg/kg)	Katahdin MDL (mg/kg)
C11-C22 AROMATICS	TTNUS152	1,800	FDEP SCTL	600	20	15	1.4
C19-C36 ALIPHATICS	TTNUS085	42,000	FDEP SCTL	14,000	20	15	0.54
C5-C8 ALIPHATICS	TTNUS083	7,100	FDEP SCTL	2,400	25	19	0.99
C9-C10 AROMATICS	TTNUS167	560	FDEP SCTL	190	25	19	0.79
C9-C12 ALIPHATICS	TTNUS168	1,700	FDEP SCTL	570	25	19	0.5
C9-C18 ALIPHATICS	TTNUS084	2,900	FDEP SCTL	970	20	15	0.79

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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: Dioxins/Furans

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQLGs (mg/kg)	CFA LOQ (mg/kg)	CFA LOD (mg/kg)	CFA MDL (mg/kg)
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.0018	EPA RSL	0.00060	0.000005	0.00000333	0.00000167
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.0018	EPA RSL	0.00060	0.000005	0.00000333	0.00000167
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.0018	EPA RSL	0.00060	0.000005	0.00000333	0.00000167
1,2,3,4,7,8-HXCDD	39227-28-6	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,4,7,8-HXCDF	70648-26-9	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,6,7,8-HXCDD	57653-85-7	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,6,7,8-HXCDF	57117-44-9	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,7,8,9-HXCDD	19408-74-3	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,7,8,9-HXCDF	72918-21-9	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
1,2,3,7,8-PECDD	40321-76-4	0.000018	EPA RSL	0.0000060	0.000005	0.00000333	0.00000167
1,2,3,7,8-PECDF	57117-41-6	0.0006	EPA RSL	0.00020	0.000005	0.00000333	0.00000167
2,3,4,6,7,8-HXCDF	60851-34-5	0.00018	EPA RSL	0.000060	0.000005	0.00000333	0.00000167
2,3,4,7,8-PECDF	57117-31-4	0.00006	EPA RSL	0.000020	0.000005	0.00000333	0.00000167
2,3,7,8-TCDD	1746-01-6	0.000007	FDEP SCTL	0.0000023	0.000001	0.000000667	0.000000333
2,3,7,8-TCDF	51207-31-9	0.00018	EPA RSL	0.000060	0.000001	0.000000667	0.000000333
OCDD	3268-87-9	0.06	EPA RSL	0.020	0.00001	0.00000666	0.00000333
OCDF	39001-02-0	0.06	EPA RSL	0.020	0.00001	0.00000666	0.00000333
TOTAL HPCDD	37871-00-4	0.00039	EPA RSL	0.00013	0.000005	0.00000333	0.00000167
TOTAL HPCDF	38998-75-3	NC	None	NC	0.000005	0.00000333	0.00000167
TOTAL HXCDD	34465-46-8	NC	None	NC	0.000005	0.00000333	0.00000167
TOTAL HXCDF	55684-94-1	NC	None	NC	0.000005	0.00000333	0.00000167
TOTAL PECDD	36088-22-9	NC	None	NC	0.000005	0.00000333	0.00000167
TOTAL PECDF	30402-15-4	NC	None	NC	0.000005	0.00000333	0.00000167
TOTAL TCDD	41903-57-5	NC	None	NC	0.000001	0.000000667	0.000000333
TOTAL TCDF	55722-27-5	NC	None	NC	0.000001	0.000000667	0.000000333

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 (FDEP, 2005); EPA-RSL: USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential (USEPA, November 2010).

Please note that data will be reported at the LOQ and MDL, with non-detected data being the MDL 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	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Draft UFP-SAP - Sites 4 and 5	Tetra Tech	01/15/2011	03/25/11	Draft UFP-SAP submittal	03/25/11
Draft Final UFP-SAP - Sites 4 and 5	Tetra Tech	05/02/11	06/03/11	Draft Final UFP-SAP submittal	06/03/11
Final UFP-SAP - Sites 4 and 5	Tetra Tech	07/11/11	07/22/11	Final UFP-SAP submittal	07/22/11
Field Team Mobilization	Tetra Tech	08/01/11	08/05/11	NA	NA
Groundwater and Soil Sampling	Tetra Tech	08/05/11	01/13/12	NA	NA
Demobilization	Tetra Tech	01/23/12	01/25/12	NA	NA
Draft SA Report - Site 4 Draft SA Report - Site 5	Tetra Tech	03/19/12	05/25/12	Report Draft Submittal	05/25/12
Draft Final SA Report - Site 4 Draft Final SA Report - Site 5	Tetra Tech	06/25/12	07/13/12	Report Draft Final Submittal	07/13/12
Final SA Report - Site 4 Final SA Report - Site 5	Tetra Tech	08/13/12	08/24/12	Report Final Submittal	08/24/12

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 4 and 5 are presented below, including the proposed sample locations, sampling methods, and a rationale for the sampling activities. The proposed sample locations for the initial phase of soil and groundwater sampling are presented on Figure 17-1. The analytical program recommended for each proposed sample is presented in Worksheet #18. The proposed groundwater and soil sampling locations for Sites 4 and 5 were chosen based on the CSM, the current understanding of site-specific conditions, and the need to collect data that will help resolve the problems described in Worksheet #11.

Samples will be collected from the vicinity of the slurry burn pit and burn kettle at Site 4 and the vicinity of the old oil pond at Site 5. These samples will be used to determine the nature of the contamination at Sites 4 and 5 and to establish COPCs. If COPCs are identified, supplemental samples will be collected at Sites 4 and 5 to delineate the extent of COPCs in site media, as required by FDEP 62-780, F.A.C.

Once the COPCs have been identified and delineated and the investigation is complete, the data will be presented in a SAR. Recommendations for Sites 4 and 5 will be based on comparing groundwater and soil data with the screening PALs identified in Worksheet #15. Based on the recommendations in the SAR, the Partnering Team will determine if it is necessary to evaluate remedial alternatives in a RAP.

Surface and Subsurface Soil Sampling

During the initial round of sampling, surface and subsurface soil data will be collected from two borings at Site 4 and from five borings at Site 5. Soil boring locations are identified on Figure 17-1, but may be relocated by the Tetra Tech FOL based on field observations, physical obstructions, or utilities. Borings will be conducted in areas that show evidence of potential release. For each boring, the soil will be collected from at least three intervals. The surface soil intervals will be collected from 0 to 6 inches bgs and from 6 inches to 2 feet. The subsurface interval will be collected from 2 to 4 feet bgs and 4 to 6 feet, or just above the water table (typically 3 to 5 feet bgs), whichever is encountered first. Due to the shallow water table, it is likely that the 4 to 6 feet bgs interval will not be collected. Soil samples will be collected using hand auger to a minimum of 4 feet in order to clear utilities. Any subsurface soil being collected at locations where permanent monitoring wells will be installed, the DPT drilling rig used to install the well will be used to collect the 4 to 6 feet bgs interval. Soil samples will be analyzed for VOCs, SVOCs (including PAHs), PCBs, metals, dioxins/furans (surface soil composite sample), TRPH, and speciated TPHs (if the TRPH results exceed the PAL). If the maximum measured concentration for any chemical exceeds its respective PAL, then it will be retained as a COPC.

In order to delineate the extent of COPCs in soils at Sites 4 and 5, a second round of sampling will be conducted. During the second round of sampling step-out borings will be collected and analyzed for the

COPCs identified in the initial round of sampling. Approximate locations of the step-out borings will be communicated to the Partnering Team through a FTMR. For each step-out boring, the soil will be collected from at least three intervals. The surface soil intervals will be collect from 0 to 6 inches bgs and from 6 inches to 2 feet. The subsurface interval will be collected from 2 to 4 feet bgs and 4 to 6 feet, or just above the water table (typically 3 to 5 feet bgs), whichever is encountered first. A FID (for VOCs) and field inspections (for visible oily residue and petroleum odors) will be used to field screen each step-out. Step-outs will be continued and submitted for laboratory analysis of COPCs until concentrations of COPCs are less than PALs. Based on the analytical results, if any COPCs are detected in the leading edge of the step-out boundary at concentrations that exceed the PAL, then additional step-out samples will be collected in additional rounds of sampling until the PALs are no longer exceeded or until facility boundaries are reached. For each interval a minimum of 10 soil samples will be collected per one quarter acre in order to evaluate the soil data according to the 95% UCL statistical approach as described in FDEP 62-780, F.A.C.

Groundwater Sampling

During the initial round of sampling, groundwater data will be collected from two newly installed permanent wells and one existing well at Site 4 and from five newly installed permanent wells and one existing well at Site 5. Monitoring well locations are identified on Figure 17-1, but proposed well locations may be relocated by the Tetra Tech FOL based on field observations, physical obstructions, or utilities. New monitoring wells will be installed in the location of the initial soil borings identified above. The groundwater samples collected during this investigation will be analyzed for VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH. If the maximum measured concentration for any chemical exceeds its respective PAL, then it will be retained as a COPC.

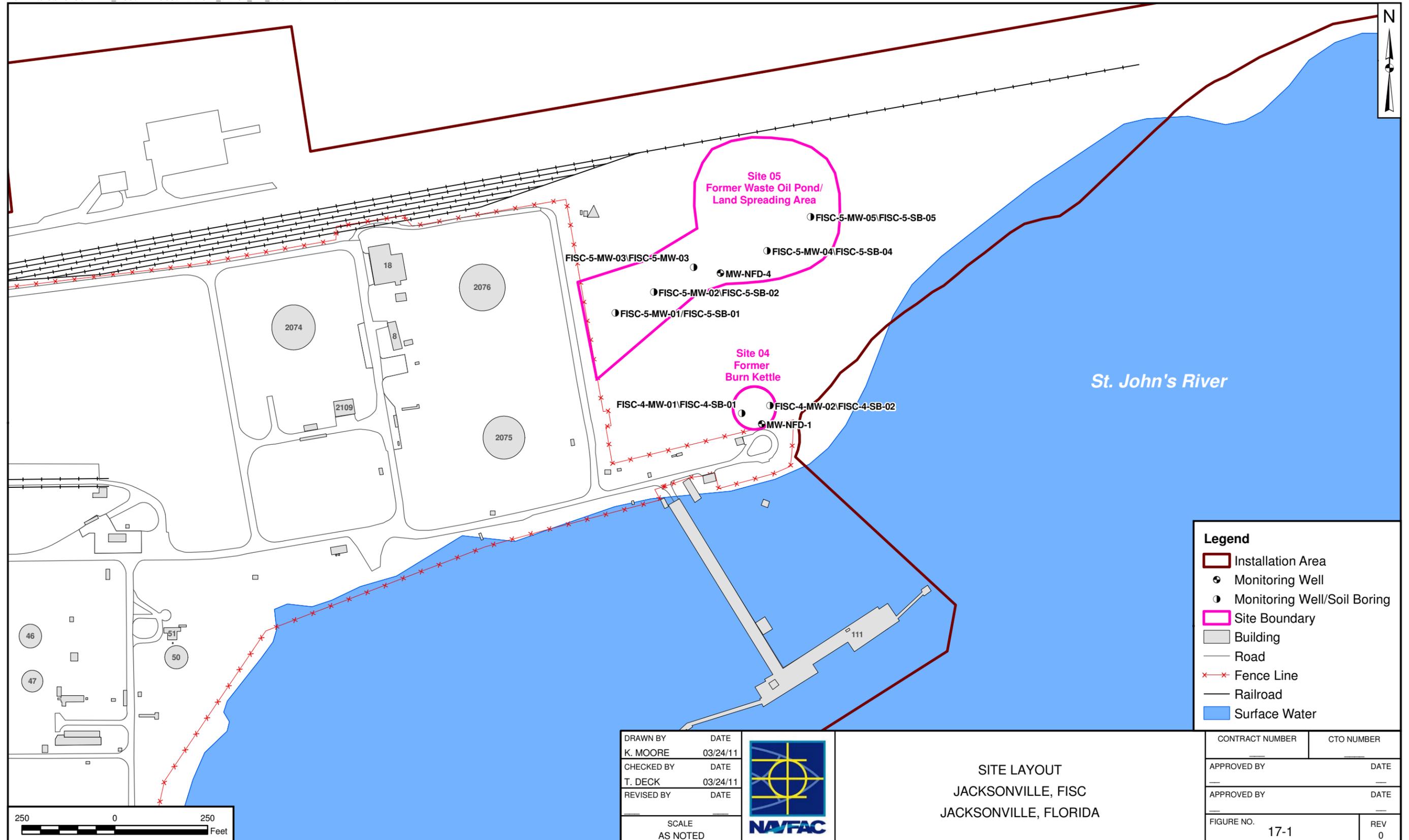
In order to delineate the extent of COPCs in groundwater at Sites 4 and 5, additional monitoring wells will be installed in a second round of sampling. It is anticipated that up to four additional permanent shallow monitoring wells at Site 4 and eight additional permanent shallow monitoring wells at Site 5 will be installed along the delineated edge of impacted soil. Approximate locations of the additional monitoring wells will be communicated to the Partnering Team through a FTMR. Each monitoring well will be sampled for the COPCs identified during the first round of the investigation. If COPCs exceed PALs in a shallow monitoring well then deep wells will be installed to vertically delineate any contamination. Based on analytical results, if any COPCs are detected at concentrations that exceed the PAL, then additional wells will be installed during an additional round of sampling until no COPCs are detected above the PAL in groundwater or until facility boundaries are reached.

General Sampling and Analysis

All surface soil, subsurface soil, and groundwater samples will be analyzed by analytical methods with sufficient sensitivity such that the results can be compared to the PALs. For samples with high concentrations, the laboratory results will be compared the FDEP practical quantitation limits (PQLs) as stated in FDEP 62-777 F.A.C.

Proposed sampling locations may be revised by the Tetra Tech FOL 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, equipment rinsate blanks, and field 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 PAHs], PCBs, dioxins/furans, 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.

PGH P:\GIS\JACKSONVILLE_FISC\MAPDOCS\MXD\FUEL_DEPOT_MW_LOCATION.MXD 3/24/11 KM



DRAWN BY	DATE
K. MOORE	03/24/11
CHECKED BY	DATE
T. DECK	03/24/11
REVISED BY	DATE

SCALE
AS NOTED



SITE LAYOUT
 JACKSONVILLE, FISC
 JACKSONVILLE, FLORIDA

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

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

Sampling Location/ID Number ⁽¹⁾	Matrix	Depth/ Location (feet bgs)	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference
SOIL SAMPLES – SITES 4 AND 5 SITE CHARACTERIZATION					
FISC-4-SB01- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-4-SB02- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-4-COMP-YYYYMMDD	Soil	0 – 0.5	Dioxin composite	1	FDEP FS 3000
FISC-5- SB01- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5- SB02- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5- SB03- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5- SB04- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5- SB05- XX-YYYYMMDD	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5-COMP-YYYYMMDD	Soil	0 – 0.5	Dioxin composite	1	FDEP FS 3000

Sampling Location/ID Number ⁽¹⁾	Matrix	Depth/ Location (feet bgs)	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference
GROUNDWATER SAMPLES – SITES 4 AND 5 SITE CHARACTERIZATION					
FISC-4-MW01-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-4-MW02-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-4-MW-NFD1-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW01-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW02-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW03-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW04-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW05-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW- NFD4-YYYYMMDD	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FIELD DUPLICATES – SOIL					
FISC-4-SB- YYYYMMDD -FD01	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5-SB- YYYYMMDD -FD01	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000
FISC-5-SB- YYYYMMDD -FD02	Soil	0 – 0.5 0.5-2 2-4 4-6	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH (and speciated TPHs if TRPH exceeds PAL)	3 or 4 ⁽²⁾	FDEP FS 3000

Sampling Location/ID Number ⁽¹⁾	Matrix	Depth/ Location (feet bgs)	Analytical Group	Number of Samples	Sampling Standard Operating Procedure Reference
FIELD DUPLICATES – GROUNDWATER					
FISC-4-MW- YYYYMMDD -FD01	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
FISC-5-MW- YYYYMMDD -FD01	Groundwater	---	VOCs, SVOCs (including PAHs), PCBs, metals, and TRPH	1	FDEP FS 2200
IDW SAMPLES (QUANTITY TO DEPEND ON NUMBER OF CONTAINERS)					
FISC-4-IDW- YYYYMMDD -01	IDW	---	Flashpoint	1	
FISC-5-IDW- YYYYMMDD -01	IDW	---	Flashpoint	1	

- XX – Sample Depth – Bottom of sample interval in feet below ground surface (bgs)
 YYYYMMDD – date of sample – year, month, date
 VOCs – volatile organic compounds
 SVOCs – semivolatiles organic compounds
 PAH- polynuclear aromatic hydrocarbons
 PCBs – polychlorinated biphenyls
 TPH – total petroleum hydrocarbons
 TRPH – total recoverable petroleum hydrocarbons IDW – investigation derived waste
- Due to the shallow groundwater at the site, the 4 to 6 feet subsurface soil interval may not be collected.

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	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram (g) Encore samplers or terracores	5 g	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	SW-846 5030/8260B/8260B SIM Empirical SOP-202	Three 40-milliliter (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	1,4-dioxane	SW-846 5030/8260B SIM Enco SOP-VGCMS-10	Three 40-milliliter (mL) glass vials	5 mL	HCl to pH<2; Cool to $\leq 6^{\circ}\text{C}$; no headspace	14 days to analysis
Soil	SVOCs	SW-846 3546/8270C Empirical SOP-201/343	One 4-ounce (oz) glass jar	15 g	Cool to $< 6^{\circ}\text{C}$	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	SVOCs	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-oz glass jar	30 g	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-L glass amber bottles	1,000 mL	Cool to $\leq 6^{\circ}\text{C}$	7 days until extraction, 40 days to analysis
Soil	Metals	SW-846 3050B/6010C Empirical SOP-100/104/105	One 4-oz glass jar	1 to 2 g	Cool to $\leq 6^{\circ}\text{C}$	180 days to analysis except mercury, 28 days for mercury
Groundwater and aqueous QC samples	Metals	SW-846 3010A/6010C Empirical SOP-100/103/105	One 500-mL plastic bottle	50 mL	Nitric acid (HNO_3) 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-oz glass jar	15 g	Cool to $\leq 6^{\circ}\text{C}$	14 days until extraction, 40 days to analysis
Soil	Speciated TPH - VPH	MADEP-VPH-04-1.1 Katahdin SOP CA-312	Two 40-mL glass vials	15 g	15 mL methanol, cool to $\leq 6^{\circ}\text{C}$	28 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)
Aqueous QC samples	Speciated TPH - VPH	MADEP-VPH-04-1.1 Katahdin SOP CA-312	Two 40-mL glass vials	40 mL	HCl to pH < 2, cool to ≤ 6 °C	14 days to analysis
Soil	Speciated TPH - EPH	MADEP-EPH-04-1.1 Katahdin SOP CA-322, CA-511	4-oz wide-mouth jar	10 g	Cool to ≤ 6 °C	14 days to extraction, 40 days to analysis
Aqueous QC samples	Speciated TPH - EPH	MADEP-EPH-04-1.1 Katahdin SOP CA-322, CA-511	Two 1-L amber glass bottles	1000 mL	5 mL of 1:1 HCl, cool to ≤ 6 °C	14 days to extraction, 40 days to analysis
Soil	Dioxins/ Furans	SW-846 8290A CFA SOP- CF-OA-E-01 and 02	Two 4 oz amber glass jars	100g	Cool to ≤ 6 °C	30 days from collection to extraction, 45 days from extraction to analysis
Aqueous QC samples	Dioxins/ Furans	SW-846 8290A CFA SOP- CF-OA-E-01 and 02	Two 1 L Glass - amber	1,000 mL	Cool to ≤ 6 °C	30 days from collection to extraction, 45 days from extraction to analysis
Soil IDW ⁴	Flashpoint	SW-846 1010A Unspecified	One 4-oz glass jar	unspecified	Cool to ≤ 6 °C	7 days to analysis
Groundwater IDW ⁴	Flashpoint	SW-846 1010A Unspecified	One 500 mL plastic bottle	unspecified	Cool to ≤ 6 °C	7 days to analysis

- 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 Matrix Spike (MS)/Matrix Spike Duplicate (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 and Groundwater 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 samples will not be validated.

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

Site	Matrix	Analytical Group	No. of Sampling Locations ¹	No. of Field Duplicates	No. of MS/MSDs ²	No. of Field Blanks ³	No. of VOC Trip Blanks	No. of Equipment Blanks ⁴	Total No. of Samples to Lab
Site 4	Soil	VOCs, SVOCs (including PAHs), PCBs, metals, TRPH, and speciated TPH	12	1	1/1	0	1 (VOCs only)	1	15
Site 4	Soil	Dioxins/ Furans	1	1	1/1	0	NA	1	3
Site 4	Groundwater	VOCs ⁵ , SVOCs (including PAHs), PCBs, metals, TRPH, and speciated TPH	3	1	1/1	0	1 (VOCs only)	1	7
Site 5	Soil	VOCs, SVOCs (including PAHs), PCBs, metals, TRPH, and speciated TPH	20	2	1/1	0	1 (VOCs only)	1	24
Site 5	Soil	Dioxins/ Furans	1	0	0/0	0	NA	0	1
Site 5	Groundwater	VOCs (including low level 1,4-dioxane) ⁵ , SVOCs (including PAHs), PCBs, metals, and TRPH	6	1	1/1	0	1 (VOCs only)	1	9

- 1 If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.
- 2 Although the matrix spike/matrix spike duplicate (MS/MSD) (or MS/sample duplicate for metals) are not typically considered field QC samples and are not included in the "Total No. of Samples to Lab", they are included here because location determination is often established in the field.
- 3 One Field Blank (source water) will be analyzed for metals only and will be submitted with groundwater samples.
- 4 Equipment Rinsate Blanks may not be necessary if the samples are collected using dedicated sampling equipment or if there is no equipment used to collect the samples.
- 5 Note that 1,4-dioxane is part of the VOC list of analytes, but groundwater samples for low level 1,4-dioxane will be analyzed at Enco and will need to be collected and shipped separately, as noted in Worksheet #19.

SAP Worksheet #21 -- Project Sampling SOP References Table
 (UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number ¹	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature (Revision 2, 03/09/09)	Tetra Tech	NA	N	Field SOPs are included in Appendix A. FDEP SOPs are also available through their website.
CT-05	Database Records and Quality Assurance (Revision 2, 01/29/01)	Tetra Tech	NA	N	
FC 1000	Cleaning/Decontamination Procedures, 2008	FDEP	NA	N	
FD1000	Documentation Procedures, 2008	FDEP	NA	N	
FM 1000	Field Mobilization Procedures, 2008	FDEP	NA	N	
FQ 1000	Field Quality Control Requirements, 2008	FDEP	NA	N	
FS 1000	General Sampling Procedures, 2008	FDEP	NA	N	
FS 2000	General Aqueous Sampling March 31, 2008 (Effective 12/3/08)	FDEP	NA	N	
FS 2200	Groundwater March 31, 2008 (Effective 12/3/08)	FDEP	NA	N	
FS 2212	Well Purging Techniques, 2008	FDEP	NA	N	
FS 2220	Groundwater Sampling Techniques, 2008	FDEP	NA	N	
FS 3000	Soil Sampling Techniques, 2008	FDEP	NA	N	
FT Series	Field Testing, 2008	FDEP	NA	N	

Reference Number	Title, Revision Date and/or Number ¹	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
GH-1.5	Borehole and Sample Logging (Revision 1, June 1999)	Tetra Tech	General field supplies	N	Field SOPs are included in Appendix A. FDEP SOPs are also available through their website.
GH-2.8	Groundwater Monitoring Well Installation (Revision 3, September 2003)	Tetra Tech	General field supplies	N	
HS-1.0	Utility Locating and Excavation Clearance (Revision 2, December, 2003)	Tetra Tech	Remote subsurface sensing, magnetometer, etc.	N	
SA-1.1	Groundwater Sample Acquisition (Revision 7, 04/07/08)	Tetra Tech	Pump and tubing, Field log book, sample log sheets, Well construction logs	N	
SA-1.3	Soil Sampling (Revision 9, 04/07/08)	Tetra Tech	Field log book, sample log sheets, boring logs	N	
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™) (Revision 3, September, 2003)	Tetra Tech	DPT Rig, accessories, and supplies	N	
SA-6.1	Non-Radiological Sample Handling (Revision 3, 03/09/09)	Tetra Tech	Field Logbook, Field Sample Forms, COCs	N	
SA-6.3	Field Documentation (Revision 3, 03/09/09)	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	

Reference Number	Title, Revision Date and/or Number ¹	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SA-7.1	Decontamination of Field Equipment (Revision 6, 01/28/09)	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	Field SOPs are included in Appendix A. FDEP SOPs are also available through their website.
GPS-1	Field Use of GPS	Tetra Tech	GPS	N	

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

Field Equipment	Activity	Frequency	Acceptance Criterion	Corrective Action	Responsible Person	SOP Reference ¹	Comments
DPT	Inspection	Daily	Equipment Inspection Sheet Criteria	Operator correction or replacement	Tetra Tech FOL or designee	FS 3000, GH-1.5, SA-2.5	None.
Disposable Hand Trowel – Soil Sampling	Inspection	Per Use	NA	Replace	Tetra Tech FOL or designee	FS 3000, GH-1.5, SA-2.5	None.
YSI 600 Series Water Quality Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Tetra Tech FOL or designee	GH-2.8, GW-001, SA-1.1, SA-1.2, Manufacturer's Guidance Manual, FDEP FT Series	To be used to determine purge completion.
Turbidity Meter (LaMotte 2020 or equivalent)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <5 Nephelometric Turbidity Unit (NTU).	Operator correction or replacement	Tetra Tech FOL or designee	GH-2.8, GW-001, SA-1.1, SA-1.2, Manufacturer's Guidance Manual, FDEP FT Series	To be used to determine purge completion.
Water Level Indicator and Oil/Water Interface Probe	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Tetra Tech FOL or designee	GH-1.2, Manufacturer's Guidance Manual	None.
Flame Ionization Detector (FID)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator correction or replacement	Tetra Tech FOL or designee	SA-1.1, SA-1.2, SA-1.3, GH-1.2, GH-1.3, GH-1.5, GH-2.8, Manufacturer's Guidance Manual	To be used to determine the potential VOC contamination in soil borings.

¹ Specify the appropriate reference letter or number from the Project Sampling SOP References Table (Worksheet #21).

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

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? 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
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
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
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	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Empirical	Y Antimony and thallium will be concentrated 4 X
Empirical SOP-201	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 PAHs)	Gas Chromatography Mass Spectrometry (GC/MS)	Empirical	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

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
Empirical SOP-211	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 (Revision 22, 07/07/10)	Definitive	Soil, groundwater, and aqueous QC samples/PCBs	Gas Chromatography Electron Capture Detector (GC/ECD)	Empirical	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	GC/MS	Empirical	N
Empirical SOP-300	GC/MS Semivolatile 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	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	N
Empirical SOP-316	Medium Level Non-Aqueous Matrix BNA & Pesticide/PCB using SW-846 Method 3550B (Revision 12, 09/09/10)	Definitive	Soil/ high concentration TRPH samples	NA/ Extraction	Empirical	Y – 5g-20 mL extraction for high concentration TRPH samples*
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	N
Empirical SOP-338	FL-PRO Method for Determination of Petroleum Range Organics (Revision 8, 04/29/10)	Definitive	Soil, Groundwater, and Aqueous Field QC Samples/ TRPH	GC/FID	Empirical	Y – 5g-20 mL sonication extraction for high concentration samples*

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
Katahdin SOP CA-312	Method for the Determination of Volatile Petroleum Hydrocarbons (MADEP - VPH) (Revision 7, 04/10)	Definitive	Soil and aqueous QC samples / Speciated TPH - VPH	GC/ FID/ Photo Ionization Detector (PID)	Katahdin	N
Katahdin SOP CA-322	Method for the Analysis of Extractable Petroleum Hydrocarbons by MADEP – EPH (Revision 9, 06/10)	Definitive	Soil and aqueous QC samples / Speciated TPH - EPH	GC/FID	Katahdin	N
Katahdin SOP CA-511	Extraction of Petroleum Hydrocarbons From Samples for Analysis by MADEP – EPH Methods (Revision 7, 04/10)	Definitive	Soil and aqueous QC samples / Speciated TPH – EPH Extraction	NA/ Extraction	Katahdin	N
CFA SOP CF-OA-E-001	Standard Operating Procedure for Dioxin/ Furan/ PCB Congener Sample Processing (Revision 3, 08/04/10)	Definitive	Soil and aqueous QC samples / Dioxins/Furans	Continuous Liquid to Liquid Extraction (CLLE)/ Soxhlet	CFA	N
CFA SOP CF-OA-E-002	Standard Operating Procedure for the Analysis of Polychlorinated Dibenzop-Dioxins and Polychlorinated Dibenzofurans (PCDDs/PCDFs) by High-Resolution Gas Chromatography/ High-Resolution Mass Spectrometry (HRGC/HRMS) (EPA SW-846 Method 8290A, EPA Method 1613B, EPA SW-846 Method 0023A) (Revision 8, 12/10)	Definitive	Soil and aqueous QC samples / Dioxins/Furans	High Resolution Gas Chromatography/ High Resolution Mass Spectrometry (HRGC/HRMS)	CFA	N
Enco SOP-VGCMS-10	Analysis of 1,4-Dioxane by GC/MS (SIM Mode) (Revision 1, 11/24/10)	Definitive	Groundwater and aqueous QC samples/ Low Level 1,4-Dioxane	GC/MS	Enco	N

¹ Copies of all the SOPs listed are included in Appendix B.

* = All samples submitted/received will be visually evaluated by field/lab personnel to determine if sample would be expected to have normal or high TRPH concentration. If high TRPH concentration is expected, the sample will be extracted for TRPH via FL-PRO using a 5g-20 mL microtip sonication. Resulting concentrations will be used to determine extraction method for SVOCs. If TRPH exceeds 10,000 mg/kg, 5g-20 mL microtip sonication will be used for SVOC extraction/analysis. If TRPH does not exceed 10,000 mg/kg, standard microwave extraction will be used. In cases where TRPH concentration is below the LOQ of 1,360 mg/kg, the 20 mL extract will be concentrated 5-fold to provide results with a nominal LOQ of 272 mg/kg – below the PAL of 340 mg/kg.

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

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ¹
GC/MS VOCs	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 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 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	CA	Person Responsible for CA	SOP Reference ¹
GC/MS VOCs	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%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
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune and before sample analysis.	Percent Drift or Difference (%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 (CA)	Person Responsible for CA	SOP Reference ¹
GC/MS SIM Low Level 1,4-Dioxane	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 %RSDs for RF of 1,4-Dioxane must be $\leq 15\%$. If not met: r^2 must be ≥ 0.990 (6 points are required for second order).	Repeat calibration if criterion is not met.	Analyst, Department Manager	Enco SOP-VGCMS-10
	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	
	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	
	ICV – Second Source	Once after each ICAL prior to sample analysis.	%R must be within 80-120%.	Correct problem and verify ICV. Reanalyze ICV and/or ICAL as appropriate.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a BFB tune.	%D must be $\leq 20\%$.	Investigate cause and repeat injection once. If failure repeats, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	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	CA	Person Responsible for CA	SOP Reference ¹
GC/MS SVOCs (including PAHs)	Breakdown Check (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	
	ICV – Second Source	Once after each ICAL prior to sample analysis	%D must be $\leq 20\%$ 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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
GC/MS SVOCs (including PAHs)	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	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenylphosphine (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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
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
	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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
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 $\leq 20\%$.	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 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
	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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
ICP-AES Metals	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	Empirical SOP-100/105
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, reprepare, 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	
	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 \geq 0.995. Option 2) r^2 must be \geq 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
GC/FID/PID Speciated TPH - VPH	ICAL	Instrument receipt, major instrument change, when Continuing Calibration Verification does not meet criteria.	The %RSD of all project compounds must be $\leq 25\%$.	Investigate and repeat ICAL.	Analyst, Department Manager	Katahdin SOP CA-312
	ICV (Second Source)	Once after each initial calibration.	The %D for all project compounds must be $\leq 25\%$ of the expected value.	(1) Reanalyze standard. (2) Reprepare standard. Reprepare standard from fresh stock.	Analyst, Department Manager	
	CCV	Analyze prior to sample analysis, after every 20 samples and at end of sequence.	The %D must be $\leq 25\%$.	Evaluate the samples: If the %D $> 25\%$ (30% for n-nonane) and sample results are $< LOQ$, narrate. Otherwise, reanalyze all samples after last acceptable CV.	Analyst, Department Manager	
GC/FID Speciated TPH - EPH	ICAL	Prior to sample analysis.	The %RSD must be $\leq 25\%$ or the r must be ≥ 0.99 .	Investigate and repeat ICAL.	Analyst, Department Manager	Katahdin SOP CA-322
	ICV (Second Source)	Immediately following calibration.	The %D for all project compounds must be $\leq 25\%$ of the expected value.	(1) Reanalyze standard. (2) Reprepare standard. Reprepare standard from fresh stock.	Analyst, Department Manager	
	CCV	After every 20 samples; If calibration curve previously analyzed, analyze daily before samples.	The %D must be $\leq 30\%$ for n-nonane and $\leq 25\%$ for all other analytes. The closing CCV may have four analytes $>$ than 25%D, but must be $< 40\%D$.	Evaluate the samples: If the %D $> 25\%$ (30% for n-nonane) and sample results are $< LOQ$, narrate. Otherwise, reanalyze all samples after last acceptable CCV.	Analyst, Department Manager	
HRGC/HRMS Dioxins/ Furans	Tune / Mass Resolution Check	At the beginning and the end of each 12-hour period of analysis.	Static resolving power must be $\geq 10,000$ (10% valley) for identified masses per method and lock-mass ion between lowest and highest masses for each descriptor and level of reference must be $\leq 10\%$ full-scale deflection.	Retune instrument and verify. Assess data for impact. If end resolution is less than 10,000, narrate or re-inject, as necessary.	Analyst, Department Manager	CFA SOP CF-OA-E-002 Sec. 14-15

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
HRGC/HRMS Dioxins/ Furans	GC Column Performance Check Solution (CPSM)	Prior to ICAL or CCV.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers must result in a valley of $\leq 25\%$ per method; <u>and</u> identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; <u>and</u> absolute retention times for switching from one homologous series to the next ≥ 10 seconds for all components of the mixture.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.	Analyst, Department Manager	CFA SOP CF-OA-E-002 Sec. 14
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Prior to sample analysis, as needed by the failure of CCV, and when a new lot is used as a standard source.	Ion abundance ratios must be within limits specified in SOP; <u>and</u> signal to noise ratio (S/N) must be $\geq 10:1$ for all target analyte ions; and RSD must be $\leq 20\%$ for RFs for all 17 unlabelled standards and 9 labeled ISS.	Correct problem, then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Department Manager	CFA SOP CF-OA-E-002 Sec. 14
	CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios must be in accordance with SOP; <u>and</u> RF (unlabelled standards) must be $\leq 20\%D$ of average RF from ICAL; <u>and</u> RF (labeled standards) must be $\leq 30\%D$ of average RF from ICAL.	Correct problem, repeat CCV. If CCV fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV <u>End of Run CCV</u> : If RF (unlabeled standards) $>20\%D$ and $\leq 25\%D$ and/or RF (labeled standards) $>30\%D$ and $\leq 35\%D$ of the average RF from ICAL, then use mean RF from bracketing CCVs to quantitate impacted samples instead of the ICAL mean RF value. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labeled), then run a new ICAL within 2 hours, and re-quantitate samples. Otherwise, reanalyze samples with positive detections.	Analyst, Department Manager	CFA SOP CF-OA-E-002 Sec. 15

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference ¹
	Qualitative Determination	Each peak to be considered a PCDD or PCDF	2,3,7,8-substituted isomers with labeled standards: RT within -1 to +3 seconds of labeled standard; 2,3,7,8-substituted isomers without labeled standards: RRT within 0.005 RRT units of that in CVS; Non-2,3,7,8-substituted isomers: RT within window definition; Ions alignment (± 2 sec.); Ion ratios in accordance with method criteria; S/N ratio of ISs ≥ 10 times background noise and S/N ratio of all remaining ions for unlabeled analytes ≥ 2.5 times background noise; and for PCDFs: no PCDFs at the same retention time as PCDF signal present having a S/N ratio ≥ 2.5 (± 2 sec). 2378-TCDF requires DB-225 confirmation when detected above the LOQ by the primary analysis.	Consider peak a nondetect when any failures occur. Consider re-extraction with less sample amount or additional cleanup where labeled compounds fail. Report estimated maximum positive concentration (EMPC) if required for failing ion ratio.	Analyst, Department Manager	CFA SOP CF-OA-E-02 Sec. 15

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

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

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	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 lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-202
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 lab Equipment Maintenance SOP.	Low Level 1,4-Dioxane	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	Enco SOP VGCMS-10
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 PAHs)	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 lab 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

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
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 SOP.	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
ICP-AES	Clean sample path, check pump tubing, argon level, vacuum and waste container daily. Clean source as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals (except mercury)	Pump, pump tubing, vacuum source, 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
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 lab 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/PID/FID	Replace or cut GC column as needed. Bake out trap and column. Change trap as needed.	Speciated TPH - VPH	Trap, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Katahdin SOP CA-312
GC/FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Speciated TPH - EPH	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, Department Manager	Katahdin SOP CA-322
HRGC/HRMS	Daily items may include septa replacement, injection port items, solvent replenishment, instrument tuning adjustment, etc.	Dioxins/ Furans	Instrument resolving power, GC performance, and isomer specificity are monitored daily.	Maintenance is ongoing and performed as needed. Preventative maintenance such as septa replacement and solvent replenishment is performed daily.	Successful daily instrument calibration per requirements.	Documentation of item addressed is located in the instruments maintenance logbook. All instrument maintenance items are recorded.	Instrument Operator	CFA SOP CF-OA-E-002

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 A)

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, Katahdin, Enco, and CFA
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical, Katahdin, Enco, and CFA
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/ Empirical, Katahdin, Enco, and CFA
Sample Determinative Analysis (Personnel/Organization): GC Lab, GC/MS Lab, Metals Lab/ Empirical; GC Lab / Katahdin; GC/MS Lab/ Enco, and HRGC/HRMS Lab/ CFA
Sample Archiving
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/ Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (No. of days from sample collection): NA
Sample Disposal
Personnel/Organization: Sample Custodians/ Empirical, Katahdin, Enco, and CFA

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. All forms must be filled in as completely as possible.

27.1.1 Sample Identification

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

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs and boring logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All 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 A.

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 PPE

27.1.3 Sample Handling and Tracking System

Following sample collection into the appropriate bottle ware, all 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 bottle ware during shipment. The cooler will be secured using strapping 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 the stages and 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 provides further details on the chain-of-custody procedure, which is provided in Appendix A.

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

27.1.4 Sample Handling

Sample handling requirements are described in Worksheet #26. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel, and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP (See Worksheet #21). 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 their integrity is preserved. Details are provided below.

27.1.5 Sample Delivery

Samples to be delivered to the laboratory will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory within 24 hours. Under no circumstances will sample holding times be exceeded.

27.1.6 Sample Custody

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

A sample is under custody if:

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

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

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

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

27.1.7 Laboratory Custody

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical, Katahdin, Enco, and CFA SOPs (Appendix B). 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 (LIMS) 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					
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	Data Quality Indicator (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.	If blank results are above $\frac{1}{2}$ LOQ (or >LOQ for common contaminants), sample results which are < LOQ or > 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-analyze all associated samples.	Analyst, Department Manager, Data Validator	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 reprepare 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 reprepared within hold time.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	VOCs					
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	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/ MSD	One per sample delivery group (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 \leq 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, Data Validator	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, Data Validator	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 \pm 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, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
Results between detection limit (DL) and LOQ	NA	Apply "J" qualifier to results detected between DL and LOQ.	NA.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	SVOCs (including PAHs)					
Analytical Method / SOP Reference	SW-846 8270C/ Empirical SOP-201					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	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, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
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, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	SVOCs (including PAHs)					
Analytical Method / SOP Reference	SW-846 8270C/ Empirical SOP-201					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD ≤30% (for LCS/LCSD, if LCSD is analyzed)	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to DOD QSM Version 4.1 Table G-1 for number of marginal xceedences allowed. Contact Client if samples cannot be reprepared within hold time.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	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, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples.	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD ≤ 30%.	CA will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager, Data Validator	Accuracy/ Bias/ Precision	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, Data Validator	Accuracy	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	CA	Person(s) Responsible for CA	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, Data Validator	Bias / Contamination	Same as QC Acceptance Limits.
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, Data Validator	Accuracy /Bias	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Blanks					
Analytical Group	TRPH					
Analytical Method / SOP Reference	FL-PRO/ Empirical SOP338					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
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 ≤ 20% (water) and ≤ 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, Data Validator	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 ≤ 20% (water) and ≤ 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, Data Validator	Accuracy/ Bias/ Precision	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	CA	Person(s) Responsible for CA	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, Data Validator	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).	If an MS/MSD was performed and is acceptable, then narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high, but the sample results are $<LOQ$, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix	%Rs should meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be $\leq 30\%$.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Department Manager, Data Validator	Accuracy / Bias / Precision	Same as QC Acceptance Limits.
Surrogates	Every field and QC sample. Two per sample: Tetrachloro-m-xylene (TCMX) Decachlorobiphenyl (DCB)	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	No CA will be taken when one surrogate is within criteria. If surrogates recoveries are high and sample is $<LOQ$, then no CA is taken. If surrogates recoveries are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Department Manager, Data Validator	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	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Results between DL and LOQ	NA.	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Department Manager, Data Validator	Accuracy	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 "J" flag if RPD >40% and discuss in the case narrative.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	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	Data Quality Indicator (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, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%R must be within 80-120% of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
Duplicate Sample	One per preparation batch of 20 or fewer samples of similar matrix.	The RPD between the original sample and duplicate should be $\leq 20\%$.	Narrate any results that are outside control limits.	Analyst, Department Manager, Data Validator	Precision	Same as QC Acceptance Limits.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS	One per 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, Data Validator	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, Data Validator	Precision	Same as QC Acceptance Limits.
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD.	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results of samples of same matrix as estimates in SDG narrative.	Analyst, Department Manager, Data Validator	Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	Speciated TPH - VPH					
Analytical Method/ SOP Reference	MADEP VPH/ Katahdin SOP CA-312					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	All target analytes must be \leq LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report samples results which are $<$ LOQ and $>10X$ the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Department Manager, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
Surrogates	Every field and QC sample. One per sample: 2,5-Dibromotoluene	Water and soil: %Rs must be within 70-130%.	Reanalyze; present both sets of data.	Analyst, Department Manager, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
LCS LCSD (not required)	One per preparation batch of 20 or fewer samples of similar matrix.	Water and soil: %Rs must be within 70-130%.	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are $<$ LOQ, narrate. Otherwise, reprepare a blank and the remaining samples.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD analyzed	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples.	Water and soil: %Rs should be within 70-130%. RPD between MS and MSD should be $\leq 50\%$.	Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. If both the LCS and MS/MSD are unacceptable, reprepare the samples and QC.	Analyst, Department Manager, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous QC Samples					
Analytical Group	Speciated TPH - EPH					
Analytical Method/ SOP Reference	MADEP EPH/ Katahdin SOP CA-322					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	All target analytes must be \leq LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report samples results which are $<$ LOQ and $>10X$ the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Department Manager, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
Surrogates	Every field and QC sample. Three per sample: ortho-Terphenyl, 5-alpha-androstane, 2-Fluorobiphenyl	Water and soil: %Rs must be within 40-140%.	Reanalyze; present both sets of data.	Analyst, Department Manager, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
LCS LCSD (not required)	One per preparation batch of 20 or fewer samples of similar matrix.	Water and soil: %Rs must be within 40-140%. RPD between LCS and LCSD must be \leq 25%, if analyzed.	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are $<$ LOQ, narrate. Otherwise, reprepare a blank and the remaining samples.	Analyst, Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples.	Water and soil: %Rs should be within 40-140%. RPD should be \leq 50%.	Evaluate the samples and associated QC: i.e. If the LCS is acceptable, narrate. If both the LCS and MS/MSD are unacceptable, reprepare the samples and QC.	Analyst, Department Manager, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.

Matrix	Soil and Aqueous QC Samples					
Analytical Group	Dioxins/Furans					
Analytical Method/ SOP Reference	SW-846 8290A/ CFA SOP CF-OA-E-002					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	One per batch of 20 or fewer samples per matrix.	All target analytes must be \leq LOQ.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Analyst, Department Manager, Data Validator	Bias / Contamination	Same as QC Acceptance Limits.
Extraction standards	Every field sample, standard and QC sample.	%Rs must be within 40-135%.	Evaluate data quality. If needed, re-extract and reanalyze the sample.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.
Ongoing Precision Recovery (OPR)	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must be within 70-130%.	Perform routine instrument maintenance. Reanalyze any associated samples.	Analyst, Department Manager, Data Validator	Accuracy / Bias Precision	Same as QC Acceptance Limits.
OPR Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must be within 70-130%. The RPD should be \leq 20%.	Perform routine instrument maintenance. Reanalyze any associated samples.	Analyst, Department Manager, Data Validator	Accuracy / Bias Precision	Same as QC Acceptance Limits.
IS	Every field sample, standard and QC sample.	The %R for each IS must be within 40-135% as per DOD QSM Version 4.1.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed target analytes, if sufficient sample material is available.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil and Aqueous QC Samples
Analytical Group	Dioxins/Furans
Analytical Method/ SOP Reference	SW-846 8290A/ CFA SOP CF-OA-E-002

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MS/MSD	One per preparatory batch of 20 or fewer 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 $\leq 20\%$.	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Analyst, Department Manager, Data Validator	Accuracy / Bias Precision	Same as QC Acceptance Limits.
Duplicate Sample (if no MSD or LCSD)	One per batch of 20 or fewer samples per matrix.	RPD between the original sample and duplicate should be $\leq 25\%$.	Evaluate parent samples. If necessary, re-extract sample and duplicate sample. Comment in case narrative.	Analyst, Department Manager, Data Validator	Precision	Same as QC Acceptance Limits.
Results between EDL and LOQ	Each sample.	Quantitated value < lower end of linear calibration range and > EDL.	Report values and flag results. Flag associated results with 'J' on Form Is.	Analyst, Department Manager, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Groundwater and Aqueous QC Samples					
Analytical Group	Low Level 1,4-Dioxane					
Analytical Method/SOP Reference	SW-846 8260C SIM/Enco SOP-VGCMS-10					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One is performed for each batch of up to 20 samples.	1,4-Dioxane must be \leq $\frac{1}{2}$ LOQ.	If blank results are above $\frac{1}{2}$ LOQ, sample results which are $<$ RL/LOQ or $>$ 10X the blank contamination concentration may be reported without corrective action. Otherwise, re-analyze all associated samples.	Analyst, Department Manager, Data Validator	Contamination/ Bias	Same as QC Acceptance Limits.
LCS	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.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are $<$ LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Department Manager, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/ 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 \leq 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, Data Validator	Accuracy / Bias/ Precision	Same as QC Acceptance Limits.
Surrogate	Every field and QC sample. Two per sample: 1,4-Difluorobenzene Toluene-d8	%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, Data Validator	Accuracy / Bias	Same as QC Acceptance Limits.
IS	Every field sample, standard, and QC sample. One per sample: 1,4-dioxane-d8	RTs for ISs must be within \pm 30 seconds and the response areas must be within -50% to +100% of the midpoint standard of the ICAL curve when ICAL is performed.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Department Manager, Data Validator	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, Data Validator	Accuracy	Same as QC Acceptance Limits.

Please note that limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

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 Jacksonville 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 Jacksonville project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Pittsburgh office.
Reports SA Report for Sites 4 and 5	All reports will be stored in hardcopy in the Tetra Tech Jacksonville 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 is tracked from its generation to its 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 Electronic Data Deliverable (NEDD) and loaded into NIRIS.

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

Matrix	Analytical Group	Sample Locations/ID 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	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 Ext. 249 brichard@empirlabs.com	NA
	SVOCs (including PAHs)	See Worksheet #18	SW-846 8270C			
	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			
Soil and Aqueous QC Samples	Speciated TPH - VPH	See Worksheet #18	MADEP-VPH-04-1.1	21 calendar days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, ME 04074 Kelly Perkins 207-874-2400 Ext. 17 kperkins@katahdinlab.com	NA
	Speciated TPH - EPH	See Worksheet #18	MADEP-EPH-04-1.1			
Soil	Dioxins/Furans	See Worksheet #18	SW-846 8290A	21 calendar days	Cape Fear Analytical, LLC 3306 Kitty Hawk Rd. Suite 120 Wilmington, NC 28405 Christopher K. Cornwell Phone 910-795-0421 Chris.Cornwell@cfanalytical.com	NA
Groundwater and Aqueous QC Samples	Low Level 1,4-Dioxane	See Worksheet #18	SW-846 8260C SIM	21 calendar days	Environmental Conservation Laboratories, Inc. 10775 Central Port Drive Orlando, FL Marcia Colon Phone: 407-826-5314 mcolon@encolabs.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 CA (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (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, Katahdin, Enco, and CFA	Laboratory QA Manager or Laboratory Manager, Empirical, Katahdin, Enco, and CFA	Laboratory QA Manager or Laboratory Manager, Empirical, Katahdin, Enco, and CFA
Laboratory System Audit ¹	Every Year	External	FDOH	FDOH (recognized NELAP Accrediting Authority)	Laboratory QA Manager or Laboratory Manager, Empirical, Katahdin, Enco, and CFA	Laboratory QA Manager or Laboratory Manager, Empirical, Katahdin, Enco, and CFA	Laboratory QA Manager or Laboratory Manager, Empirical, Katahdin, Enco, and CFA

¹ Empirical, Katahdin, and CFA are DoD ELAP and FDOH NELAP accredited for all analytical groups and target analytes required for this project. Enco is FDOH NELAP accredited for low level 1,4-dioxane analysis. The DoD ELAP and FDOH NELAP accreditation letters are included in Appendix B.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses Table
 (UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Marcia McGinnity, Laboratory QAM, Empirical Leslie Dimond, Laboratory QAM, Katahdin Mike Larkins, Laboratory QAM, CFA Lori Mangrum, Laboratory QAM, Enco	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 Leslie Dimond, Laboratory QAM, Katahdin Mike Larkins, Laboratory QAM, CFA Lori Mangrum, Laboratory QAM, Enco	Specified by NELAP	Letter	FDOH	Specified by NELAP

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

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

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, Katahdin, Enco, and CFA	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, Katahdin, Enco, and CFA 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, Katahdin, Enco, and CFA
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, Katahdin, Enco, and CFA

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

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

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 are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36.	
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, Low Level 1,4-Dioxane, SVOCs (including PAHs), PCBs, and TRPH	100% limited validation* will be performed using criteria for SW-846 Methods 8260B, 8260C SIM, 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	Metals (including mercury)	100% limited 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
IIa and IIb	Soil and Aqueous Field QC Samples	Speciated TPH - VPH and EPH	100% limited validation* will be performed using criteria for Methods MADEP-VPH-04-1.1 and MADEP-EPH-04-1.1 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

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil	Dioxins/ Furans	100% limited validation* will be performed using criteria for SW-846 Method 8290A and those criteria listed in Worksheets #12, #15, #24, and #28, and the current DoD QSM. The logic outlined in "USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review", (USEPA, September 2005) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

*- Limited data validation focuses the review on specific review parameters (Data Completeness/Data Verification, Holding times, Calibrations, Blank Contamination, and Detection Limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal report, similar to a full data validation report, is prepared but the scope is more limited than a full validation report. The data packages provided by Empirical will be expansive enough to allow future complete formal data validation, if necessary.

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

Data Usability Assessment

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

Completeness

- For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Partnering 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 they do, the Tetra Tech PM will consult with the Navy RPM and other Partnering 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 Partnering 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 Partnering 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 LCS. 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 Partnering 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 Partnering Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias

among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Tetra Tech Project Chemist indicates that such quantitative analysis is required.

Sensitivity

- The Tetra Tech Project Chemist acting on behalf of the Partnering 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 Partnering Team members identified by the Tetra Tech PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical 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, 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 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.

REFERENCES

DoD (United States Department of Defense), 2009. *Department of Defense Quality Systems Manual for Environmental Laboratories*. Version 4.1. April.

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APPENDIX A

FIELD STANDARD OPERATING PROCEDURES AND FIELD FORMS



Tetra Tech NUS, Inc.

OVERBURDEN MONITORING WELL SHEET FLUSH - MOUNT

WELL NO.: _____

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	DEVELOPMENT METHOD _____
FIELD GEOLOGIST _____		
GROUND ELEVATION _____	DATUM _____	

ACAD:FORM_MWFM.dwg 07/20/99 INL

FLUSH MOUNT
SURFACE CASING
WITH LOCK



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

TYPE OF PROTECTIVE CASING: _____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

ELEVATION/DEPTH TOP OF SEAL: _____ /

TYPE OF SEAL: _____

ELEVATION/DEPTH TOP OF SAND: _____ /

ELEVATION/DEPTH TOP OF SCREEN: _____ /

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ /

ELEVATION / DEPTH BOTTOM OF SAND: _____ /

ELEVATION/DEPTH BOTTOM OF HOLE: _____ /

BACKFILL MATERIAL BELOW SAND: _____

Standard Operating Procedure for Investigative Derived Waste

1. At Naval Station Mayport (NAVSTA), Investigative Derived Waste is defined as soil or water that is generated from the remedial investigation of contaminated sites. IDW can include, but not be limited to, drill cuttings, purge water, soil, sediment or decontamination water. Operations usually associated with IDW include soil and groundwater sampling, monitoring well installation and decontamination of equipment used for sampling and installation.
2. IDW will be containerized when generated and kept at the site of generation as coordinated with the tenant occupying the area. Drums can be moved to other locations in the general area to accommodate NAVSTA personnel movement or requirements within reason. A central location can be identified prior to the sampling event if in the best interest of the government.
3. IDW drums shall be clearly identified with "Awaiting Analytical" sticker visible containing contractor name and phone number, generation location, date of generation, NAVSTA point of contact, and contents of drum. A drum log using the format of Enclosure (1) shall be completed for each drum and provided to the NAVSTA point of contact when drum is generated. Drums shall be inspected weekly until disposal using Enclosure (2) and inspection form shall be faxed to NAVSTA Environmental Department. When sample results have been received, the analytical shall be provided to the NAVSTA point of contact for waste and disposal determination. The contractor shall be responsible for disposal of all IDW. IDW with analytical results less than Cleanup Target Levels identified in 62-777 Florida Administrative Code may be disposed onsite if sufficient soil is at location. IDW may not be disposed in storm drain or on an impervious surface. In certain conditions, non-hazardous IDW may be disposed through a sewer lift station to the Wastewater Treatment Plant with prior written approval by the Utility Engineer at Public Works Center Jacksonville.
4. If the IDW is identified as hazardous waste, the contractor shall manage drums per the NAVSTA Hazardous Waste Management Plan (SOPA(ADMIN) MYPTINST 5090.1F) and shall be disposed through the NAVSTA Hazardous Waste Storage Facility with the contractor paying disposal cost to PWC (2005 cost approximately \$1.75/pound). IDW that is not hazardous waste but does not meet the Target Levels to be disposed onsite, the contractor shall arrange for the IDW to be legally transported and disposed at an approved facility. The contractor will coordinate with NAVSTA personnel to sign the non-hazardous manifest as generator.

Naval Station Mayport Investigative Derived Waste Drum Log

Contractor Company Name: _____

Individual Name: _____

Location Name: _____
(i.e. SWMU number, Bldg number)

Date of generation: _____

Expected date of results: _____

Drum Number: _____
(Use site # and unique drum number)

<u>Type of Waste</u> (i.e. drill cuttings, purge water)	<u>Quantity of Waste</u> (gals/lbs)	<u>Date</u>	<u>Individual's Initials/ Name</u>

Enclosure (1)

WEEKLY INVESTIGATIVE DERIVED WASTE INSPECTION CHECKLIST
NAVAL STATION MAYPORT

This form is to be completed legibly by the contractor when conducting weekly inspections of IDW drums.

All discrepancies shall be corrected immediately. Failure to correct discrepancy(s) shall result in contractual action.

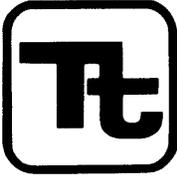
Date: _____

Inspector: _____

Company Name: _____

		YES	NO
1.	Are all containers properly labeled/dated?		
2.	Are containers compatible with contents?		
3.	Are all containers in good condition?		
4.	Are containers closed?		
5.	Are lids/caps/bolts/rings tight?		
6.	Are any containers dated longer than 60 days?		
7.	Number of containers inspected. _____		
Comments:			
Date/nature of repairs or remedial actions:			
Copy to: NAVSTA Mayport N4E FAX: 270-7398 (EACH FRIDAY)			

Enclosure (2)



TETRA TECH NUS,
INC.

STANDARD OPERATING PROCEDURES

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

Subject
SAMPLE NOMENCLATURE

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1.0 PURPOSE

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

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

2.0 SCOPE

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

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

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

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

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

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

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

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5.0 PROCEDURES

5.1 INTRODUCTION

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

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

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

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

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

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

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

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

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

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

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

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

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

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

Sample type - Examples of sample types are as follows:

- AH - Ash Sample

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

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

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

Species identifier - Examples of species identifier are as follows:

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

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

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

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

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

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A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

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

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

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

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

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

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

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.

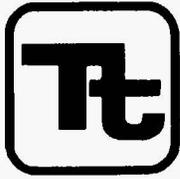
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The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

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.



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

Subject
DATABASE RECORDS AND QUALITY ASSURANCE

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

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

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

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

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

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

4.0 RESPONSIBILITIES

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

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

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

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

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

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

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

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

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

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

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

5.0 PROCEDURES

5.1 Introduction

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

5.2 File Establishment

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

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

5.3 Electronic Deliverables

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

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5.4 Sample Tracking Forms

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

5.5 Chain-of-Custody Forms

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

5.6 Data Validation Letters

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

5.7 Historical Data

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

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

6.0 RECORDS

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

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

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

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ATTACHMENT A



MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name:	Request Date:
CTO:	Date Data Available for Production:
Project Manager:	Request in Support of:
Requestor:	Database Lead:
Program/Client:	GIS Lead:
State/EPA Region:	Statistics Lead:
	Risk Lead:
Site Name(s) (Area, OU, etc.):	
Sampling Date(s):	
Matrix: <input type="checkbox"/> GW <input type="checkbox"/> SO <input type="checkbox"/> SD <input type="checkbox"/> SW <input type="checkbox"/> Other:	
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).
- 4). Check manufacturer's instructions for cleaning restrictions and/or recommendations.
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 for extended 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.

- 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 at wash cycle, using laboratory detergent cycle, followed by tap and analyte-free water rinse cycles.
 - 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 that utilize in-line, molded and disposable filters.
2. Dissolved Constituents using Non-disposable 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

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 return to the vehicle for cleaning or to transport all cleaning materials to the staging location, at a minimum thoroughly rinse the pump with water.

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

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- 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

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

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 relative coverage of any oil on the water surface
- Term that best describes the amount 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 score
 - 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

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 collected
- Algal thickness rank (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 analysts collecting and sorting samples
- Habitat types (substrates) sampled
- 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 grab dredge sample for each drop (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: _____	
<p>DECONTAMINATION SUPPLIES</p> <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: _____	<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	<input type="checkbox"/> GPS equipment <input type="checkbox"/> Calculator <p>REFERENCE MATERIALS</p> <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 <p>HEALTH & SAFETY SUPPLIES</p> <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
<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	<p>SAMPLE TRANSPORTATION SUPPLIES</p> <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	<p>FIELD MEASUREMENT EQUIPMENT</p> <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
<p>PRESERVATION SUPPLIES</p> <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	<p>DOCUMENTATION SUPPLIES</p> <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	

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
 - Stainless steel
 - Polyethylene
 - 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 composite sampler
 - Other
- Tubing
- Teflon™ _____ Sets
 - Polyethylene _____ Sets
 - Polypropylene _____ Sets
 - Vinyl _____ Sets
 - Rubber _____ Sets
 - Tygon _____ Sets

- Bailers
- Teflon
 - Stainless Steel
 - 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 _____ Sets
 - Polyethylene _____ 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
 - Stainless steel

- SEDIMENTS**
- Dredges
- Petersen
 - Ponar
 - Ekman
 - Young Grab
 - Van Veen
 - 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:	
<p>WASTE</p> <input type="checkbox"/> Stainless steel scoop <input type="checkbox"/> Stainless steel spoons or spatulas <input type="checkbox"/> Stainless steel push tubes <input type="checkbox"/> Stainless steel auger <input type="checkbox"/> Stainless steel Ponar dredge <input type="checkbox"/> Glass coliwasa <input type="checkbox"/> Drum thief <input type="checkbox"/> Mucksucker <input type="checkbox"/> Dipstick <input type="checkbox"/> Stainless steel bacon bomb <input type="checkbox"/> Stainless steel bailer <input type="checkbox"/> Teflon bailer <input type="checkbox"/> Peristaltic pump <input type="checkbox"/> Stainless steel split spoon <input type="checkbox"/> Roto-hammer <input type="checkbox"/> Glass tubing <p>SHELLFISH</p> <input type="checkbox"/> Seine <input type="checkbox"/> Trawl <input type="checkbox"/> Bucket type/double pole <input type="checkbox"/> Tong/Double handed grab <input type="checkbox"/> Line or cable operated grab bucket <input type="checkbox"/> Petersen <input type="checkbox"/> Ponar <input type="checkbox"/> Ekman <input type="checkbox"/> Orange-peel grab <input type="checkbox"/> Biological or hydraulic dredge <input type="checkbox"/> Scoops/shovels <input type="checkbox"/> Scrapers <input type="checkbox"/> Rakes <input type="checkbox"/> D-traps <p>Processing Equipment</p> <input type="checkbox"/> Holding trays <input type="checkbox"/> Stainless steel shucking knife <input type="checkbox"/> Calipers or ruler <input type="checkbox"/> Aluminum foil <input type="checkbox"/> Plastic bags <p>FINFISH</p> <input type="checkbox"/> Electrofishing devices <input type="checkbox"/> Seines	<input type="checkbox"/> Trawls <input type="checkbox"/> Angling <input type="checkbox"/> Gill net <input type="checkbox"/> Trammel net <input type="checkbox"/> Hoop, fyke & pound nets <input type="checkbox"/> D-traps <p>Processing Equipment</p> <input type="checkbox"/> Holding trays <input type="checkbox"/> Measuring board or ruler <input type="checkbox"/> Stainless steel descaler <input type="checkbox"/> Stainless steel scalpel <input type="checkbox"/> Balance <input type="checkbox"/> Aluminum foil <input type="checkbox"/> Plastic bags <p>BIOLOGICAL COMMUNITY SAMPLING</p> <p>Phytoplankton</p> <input type="checkbox"/> Van Dorn <input type="checkbox"/> Alpha bottle <input type="checkbox"/> Logol's solution <p>Periphyton</p> <input type="checkbox"/> Periphytometer <input type="checkbox"/> Microscope slides <input type="checkbox"/> 100% buffered formalin <input type="checkbox"/> Nylon twine <p>Qualitative Periphyton Sampling</p> <input type="checkbox"/> Stainless steel spatula/spool <input type="checkbox"/> Stainless steel forceps <input type="checkbox"/> Suction bulb <input type="checkbox"/> Preservative <input type="checkbox"/> Buffered formalin <input type="checkbox"/> Lugol's solution <input type="checkbox"/> M3 <input type="checkbox"/> Resealable plastic bags <input type="checkbox"/> White picking pan <p>Benthic Macroinvertebrates</p> <input type="checkbox"/> Forceps <input type="checkbox"/> Transfer pipettes <input type="checkbox"/> White picking pans <input type="checkbox"/> 10X hand lens <input type="checkbox"/> Alcohol-filled jars <input type="checkbox"/> Dip net (30 mesh) <input type="checkbox"/> Hester-Dendy <input type="checkbox"/> Coring device	<input type="checkbox"/> Dredge <input type="checkbox"/> Ekman <input type="checkbox"/> Petite ponar <input type="checkbox"/> 30 mesh box sieve

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.

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:

DEP-SOP-001/01
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 1000. GENERAL SAMPLING PROCEDURES

See also the following Standard Operating Procedures:

- FA 1000 and 2000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

FS 1001. Preliminary Activities

1. Begin each sampling trip with some planning and coordination. Refer to FM 1000 for recommendations and suggestions on laboratory selection and communication, and field mobilization.

1.1. DEP recommends that a minimum of two people be assigned to a field team. In addition to safety concerns, the process of collecting the samples, labeling the containers and completing the field records is much easier if more than one person is present.

1.2. If responding to incidents involving hazardous substances, DEP recommends that four or five people be assigned to the team.

2. EQUIPMENT

2.1. Select appropriate equipment based on the sampling source (see FS 2000 to FS 8200), the analytes of interest and the sampling procedure.

2.1.1. If properly cleaned, sample containers may be used as collection devices or intermediate containers.

2.2. The equipment construction must be consistent with the analytes or analyte groups to be collected (see Tables FS 1000-1 and FS 1000-2).

2.3. Bring precleaned equipment to the field or use equipment that has been certified clean by the vendor or laboratory.

3. DEDICATED EQUIPMENT STORAGE

3.1. Store all dedicated equipment (except dedicated pump systems or dedicated drop pipes) in a controlled environment.

3.2. If possible, store equipment in an area that is located away from the sampling site. If equipment other than dedicated pumps or dedicated drop pipes is stored in monitoring wells, suspend the equipment above the formation water.

3.3. Securely seal the monitoring well in order to prevent tampering between sampling events.

3.4. Decontaminate all equipment (except dedicated pumps or drop pipes) before use according to the applicable procedures in FC 1000.

4. SAMPLE CONTAINERS

4.1. The analyses to be performed on the sample determine the construction of sample containers.

4.2. Inspect all containers and lids for flaws (cracks, chips, etc.) before use. Do not use any container with visible defects or discoloration.

FS 1002. *Contamination Prevention and Sample Collection Order*

1. CONTAMINATION PREVENTION

1.1. Take special effort to prevent cross contamination and contamination of the environment when collecting samples. Protect equipment, sample containers and supplies from accidental contamination.

1.1.1. Do not insert pump tubing, measurement probes, other implements, fingers, etc. into sample containers or into samples that have been collected for laboratory analysis.

1.1.1.1. If it is necessary to insert an item into the container or sample, ensure that the item is adequately decontaminated for the analytes of interest to be analyzed in the sample.

1.1.2. If possible, collect samples from the least contaminated sampling location (or background sampling location) to the most contaminated sampling location.

1.1.2.1. Collect the ambient or background samples first and store them in separate ice chests or shipping containers.

1.1.3. Collect samples in flowing water from downstream to upstream.

1.1.4. Do not store or ship highly contaminated samples (concentrated wastes, free product, etc.) or samples suspected of containing high concentrations of contaminants in the same ice chest or shipping container with other environmental samples.

1.1.4.1. Isolate these sample containers by sealing them in separate, untreated plastic bags immediately after collecting, preserving, labeling, etc.

1.1.4.2. Use a clean, untreated plastic bag to line the ice chest or shipping container.

2. SAMPLE COLLECTION ORDER

2.1. Sampling order is a recommendation to be modified depending on site circumstances. Unless field conditions justify other sampling regimens, collect samples in the following order:

- Volatile Organics and Volatile Inorganics
- Extractable Organics, Petroleum Hydrocarbons, Aggregate Organics and Oil & Grease
- Total Metals
- Dissolved Metals
- Inorganic Nonmetallics, Physical and Aggregate Properties, and Biologicals
- Radionuclides
- Microbiological

Note: If the pump used to collect groundwater samples cannot be used to collect volatile or extractable organics, then collect all other parameters, withdraw the pump and tubing, and collect the volatile and extractable organics.

3. COMPOSITE SAMPLES

- 3.1. Do not collect composite samples unless required by permit or DEP program.
- 3.2. If compositing is required, use the following procedure:
 - 3.2.1. Select sampling points from which to collect each aliquot.
 - 3.2.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.
 - 3.2.3. Record the approximate amount of each aliquot (volume or weight).
 - 3.2.4. Add preservative(s), if required.
 - 3.2.5. Label container and make appropriate field notes (see FD 1000-9000).
 - 3.2.6. Notify the laboratory that the sample is a composite sample.
 - 3.2.7. When collecting soil or sediment samples, combine the aliquots of the sample directly in the sample container with no pre-mixing. Notify the laboratory that the sample is an unmixed composite sample, and request that the laboratory thoroughly mix the sample before sample preparation or analysis.
 - 3.2.8. When collecting water composites see FS 2000, section 1.3 or pertinent sections of other water matrix SOPs for specific details on collection.

FS 1003. *Protective Gloves*

1. Gloves serve a dual purpose to:
 - Protect the sample collector from potential exposure to sample constituents
 - Minimize accidental contamination of samples by the collector
2. The DEP recommends wearing protective gloves when conducting all sampling activities. They must be worn except when:
 - The sample source is considered to be non-hazardous
 - The samples will not be analyzed for trace constituents
 - The part of the sampling equipment that is handled without gloves does not contact the sample source
3. Do not let gloves come into contact with the sample or with the interior or lip of the sample container.
4. Use clean, new, unpowdered and disposable gloves.
 - 4.1. DEP recommends latex gloves, however, other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.
 - 4.2. Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. Many vendor catalogs provide information about the permeability of different gloves and the circumstances under which the glove material might be applicable.
 - 4.3. The powder in powdered gloves can contribute significant contamination and DEP does not recommend wearing powdered gloves unless it can be demonstrated that the powder does not interfere with the sample analysis.

5. If gloves are used, change:
 - After preliminary activities such as pump placement;
 - After collecting all the samples at a single sampling point; or
 - If torn, or used to handle extremely dirty or highly contaminated surfaces.
6. Properly dispose of all used gloves.

FS 1004. *Container and Equipment Rinsing*

When collecting aqueous samples, rinse the sample collection equipment with a portion of the sample water before taking the actual sample. Sample containers do not need to be rinsed. In the case of petroleum hydrocarbons, oil & grease or containers with premeasured preservatives, the sample containers cannot be rinsed.

FS 1005. *Fuel-Powered Equipment and Related Activities*

1. Place all fuel-powered equipment away from, and downwind of, any site activities (e.g., purging, sampling, decontamination). If field conditions preclude such placement (i.e., the wind is from the upstream direction in a boat), place the fuel source(s) as far away as possible from the sampling activities and describe the conditions in the field notes.
2. Handle fuel (i.e., filling vehicles and equipment) prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves. Dispense all fuels, dispose of gloves downwind, and well away from the sampling activities.
3. If sampling at active gas stations, stop sample collection activities during fuel deliveries.

FS 1006. *Preservation, Holding Times and Container Types*

1. Preserve all samples according to the requirements specified in Tables FS 1000-4 through FS 1000-10.
 - 1.1. The information listed in the above-referenced tables supersedes any preservation techniques, holding time or container type that might be discussed in individual analytical methods.
 - 1.2. If samples are collected only for total phosphorus and are not for NPDES compliance, thermal preservation (ice) is not required if the sample containers are pre-preserved with acid.
2. The preservation procedures in the referenced tables specify immediate preservation. "Immediate" is defined as "within 15 minutes of sample collection." Perform all preservation on-site (in the field).
 - 2.1. Preservation is not required if samples can be transported back to the laboratory within 15 minutes of collecting the sample and
 - 2.1.1. The laboratory begins sample analysis within the 15-minute window and documents the exact time the analysis began, or
 - 2.1.2. The laboratory adds the appropriate preservatives (including thermal preservation) within 15 minutes of sample collection and documents the exact time that the preservation was done.
3. PRESERVING COMPOSITE WATER SAMPLES

3.1. If the sample preservation requires thermal preservation (e.g., $<6^{\circ}\text{C}$), the samples must be cooled to the specified temperature.

3.1.1. Manually collected samples to be composited must be refrigerated at a temperature equal to or less than the required temperature.

3.1.2. Automatic samplers must be able to maintain the required temperature by packed ice or refrigeration.

3.2. When chemical preservation is also required, begin the preservation process within 15 minutes of the last collected sample.

3.3. Holding Times for Automatic Samplers:

3.3.1. If the collection period is 24 hours or less, the holding time begins at the last scheduled sample collection;

3.3.2. If the collection period exceeds 24 hours, the holding time begins with the time that the first sample is collected.

4. PH ADJUSTED PRESERVATION - Check the pH of pH-adjusted samples according to these frequencies:

4.1. During the first sampling event at a particular site, check **all** samples (includes each groundwater monitoring well, surface water location, or influent/effluent sampling location) that are pH-adjusted except volatile organics.

4.2. During subsequent visits to a particular site, check at least one sample per parameter group that must be pH-adjusted.

4.3. If the frequency of sample collection at a specified location is greater than once per month (i.e., weekly or daily), check the pH of at least one sample per parameter group (except volatile organics) according to the following schedule:

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

4.4. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.

4.5. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

5. THERMAL PRESERVATION

5.1. When preservation requirements indicate cooling to a specific temperature, samples must be placed in wet ice within 15 minutes of sample collection (see 1006, section 2 above). Unless specified, do not freeze samples.

5.2. All supplies (ice, dry ice, etc.) necessary to meet a thermal preservation requirement must be onsite for immediate use.

5.3. Ship samples in wet ice. If samples are cooled to the required temperature before shipment, samples may be shipped with frozen ice packs if the specified temperature is maintained during shipment. The sample temperature must not exceed the specified temperature.

5.4. If immediate freezing is required, dry ice must be available in the field to begin the freezing process.

FS 1007. *Preventive and Routine Maintenance*

Preventive maintenance activities are necessary to ensure that the equipment can be used to obtain the expected results and to avoid unusable or broken equipment while in the field.

Equipment is properly maintained when:

- It functions as expected during mobilization; and
- It is not a source of sample contamination (e.g., dust).

1. Follow the manufacturer's suggested maintenance activities and document all maintenance. At a minimum, DEP recommends the activities listed on Table FS 1000-12.

2. Maintain documentation for the following information for each piece of equipment or instrumentation. See FD 3000 also.

2.1. 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.

2.2. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures and solution or parts replacement for instrument probes.

2.3. Include the calendar date for the procedures performed.

2.4. Record names of personnel performing the maintenance or repair tasks.

2.5. Describe any malfunctions necessitating repair or service.

2.6. Retain vendor service records for all affected instruments.

2.7. Record the following for rented equipment:

- Rental date(s)
- Equipment type and model or inventory number or other description

2.8. Retain the manufacturer's operating and maintenance instructions.

FS 1008. *Documentation and References*

1. REFERENCES: All sampling references must be available for consultation in the field. These include:

- DEP SOPs;
- Internal SOPs;
- Sampling and analysis plans; and/or
- Quality Assurance Project Plans.

2. DOCUMENTATION: Complete and sign all documentation (see FD 1000).

FS 1009. *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

1.1. Document all activities related to a sampling event, including sample collection, equipment calibration, equipment cleaning and sample transport.

1.2. The required documentation related to each sampling or other field activity is specified in the associated SOPs; i.e., FQ 1000, FC 1000, the FS series, and the FT series.

1.3. The documentation requirements are also summarized in FD 1000, Field Documentation. FD 1000 additionally contains a list of example forms published with the SOPs that may be used to document various activities or as templates for creating customized forms.

2. LEGAL CHAIN OF CUSTODY (COC)

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 unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

Evidentiary sample custody protocols are used to demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

When a client or situation requires legal COC, use the procedures in FD 7000 to document and track all time periods associated with the physical possession and storage of sample containers, samples, and subsamples from point of origin through the final analytical result and sample disposal.

When legal or evidentiary COC is required, samples must be:

- In the actual possession of a person who is authorized to handle the samples (e.g., sample collector, laboratory technician);
- In the view of the same person after being in their physical possession;
- Secured by the same person to prevent tampering; or
- Stored in a designated secure area.

2.1. 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.

2.1.1. Limit the number of individuals who physically handle the samples as much as practicable.

2.1.2. 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.

2.1.3. 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.

2.1.4. 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. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

2.2. Use a Chain of Custody form or other transmittal record to document sample transfers to other parties. Other records and forms may be used to document internal activities if they meet the requirements for legal chain of custody.

2.3. Legal COC begins when the precleaned sample containers are dispatched to the field.

2.3.1. The person who relinquishes the prepared sample kits or containers and the individual who receives the sample kits or containers must sign the COC form unless the same party provides the containers and collects the samples.

2.3.2. All parties handling the empty sample containers and samples are responsible for documenting sample custody, including relinquishing and receiving samples, except commercial common carriers.

2.4. Shipping Samples under Legal COC

2.4.1. Complete all relevant information on the COC transmittal form or record (see FD 7200, section 2).

2.4.2. Internal records must document the handling of the samples and shipping containers in preparation for shipment. The names of all persons who have prepared the shipment must be recorded. All time intervals associated with handling and preparation must be accounted for.

2.4.3. Place the forms in a sealed waterproof bag and place in the shipping container with the samples.

2.4.4. Seal the shipping container with tamper-proof seals (see 2.6 below) so that any tampering can be clearly seen by the individual who receives the samples.

2.4.5. Note: The common carrier does not sign COC records. However, the common carrier (when used) must be identified.

2.5. Delivering Samples to the Laboratory

2.5.1. All individuals who handle and relinquish the sample containers must sign the transmittal form. The legal custody responsibilities of the field operations end when the samples are relinquished to the laboratory.

2.6. 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.

2.6.1. Place the seal so that the closure cannot be opened without breaking the seal.

2.6.2. Record the time, calendar date and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.

2.6.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FS 1010. *Health and Safety*

Implement all local, state and federal requirements relating the health and safety.

FS 1011. *Hazardous Wastes*

Follow all local, state and federal requirements pertaining to the storage and disposal of any hazardous or investigation-derived wastes.

1. Properly manage all investigation-derived waste (IDW) so contamination is not spread into previously uncontaminated areas.
 - 1.1. IDW includes all water, soil, drilling mud, decontamination wastes, discarded personal protective equipment (PPE), etc. from site investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, and abandonment, and other investigative activities. Containerize the IDW at the time it is generated.
 - 1.2. Determine if the IDW must be managed as Resource Conservation and Recovery Act (RCRA) regulated hazardous waste through appropriate testing or generator knowledge. Manage all IDW that is determined to be RCRA regulated hazardous waste according to the local state and federal requirements.
 - 1.3. Properly dispose of IDW that is not a RCRA-regulated hazardous waste but is contaminated above the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality.
 - 1.4. IDW that is not contaminated or contains contaminants below the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality may be disposed of onsite as long as the IDW will not cause a surface water violation.
 - 1.5. Maintain all containers holding IDW in good condition:
 - 1.5.1. Periodically inspect the containers for damage
 - 1.5.2. Ensure that all required labeling (DOT, RCRA, etc.) are clearly visible.

Appendix FS 1000
Tables, Figures and Forms

- Table FS 1000-1 Equipment Construction Materials
- Table FS 1000-2 Construction Material Selection for Equipment and Sample Containers
- Table FS 1000-3 Equipment Use and Construction
- Table FS 1000-4 40 CFR Part 136 Table II: Required Containers, Preservation Techniques, and Holding Times (Water/Wastewater Samples)
- Table FS 1000-5 Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times for Analytes not found in 40 CFR Part 136
- Table FS 1000-6 Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples.
- Table FS 1000-7 Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035
- Table FS 1000-8 Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II
- Table FS 1000-9 Containers, Preservation and Holding Times for Biosolids Samples and Protozoans
- Table FS 1000-10 Container Materials, Preservation, and Holding Times for Fish and Shellfish
- Table FS 1000-11 Holding Times for SPLP or TCLP Extraction, Sample Preparation and Determinative Analysis
- Table FS 1000-12 Preventive Maintenance Tasks
- Figure FS 1000-1 Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump

DEP-SOP-001/01
 FS 1000 General Sampling Procedures
Table FS 1000-1
Equipment Construction Materials

Construction Material ¹	Acceptable Analyte Groups	Precautions
Metals		
316 Stainless Steel	All analyte groups. Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted. ²
300-Series Stainless Steel (304, 303, 302)	Suitable for all analyte groups (if used, check for corrosion before use). Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted. ² If corroded, there is a potential for samples to be contaminated with iron, chromium, copper or nickel. Check for compatibility with water chemistry for dedicated applications. Do not use in low pH, high chloride, or high TDS waters.
Low Carbon Steel Galvanized Steel Carbon Steel	Inorganic nonmetallics only.	Coring devices are acceptable for all analyte groups if appropriate liners are used. Use Teflon liners for organics. Use plastic or Teflon liners for metals. Do not use if weathered, corroded or pitted. ² If corroded, there is a potential for samples to be contaminated with iron and manganese. Galvanized equipment will also contaminate with zinc and cadmium. If used to collect large samples (e.g., dredges), collect organic and metal samples may be collected from portions of the interior of the collected material.
Brass	Inorganic nonmetallics only.	Do not use if weathered, corroded or pitted. ²
Plastics ³		
Teflon and other fluorocarbon polymers	All analyte groups. Especially recommended for trace metals and organics.	Easily scratched. Do not use if scratched or discolored.
Polypropylene Polyethylene (All Types)	All analyte groups.	Easily scratched. Do not use if scratched or discolored.
Polyvinyl chloride (PVC)	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organics samples.

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Table FS 1000-1
Equipment Construction Materials

Construction Material ¹	Acceptable Analyte Groups	Precautions
Tygon, Silicone, Neoprene	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organic samples. Do not use silicone if sampling for silica.
Viton	All analyte groups except extractable and volatile organics. ⁴	Minimize contact with sample. Use only if no alternative material exists.
Glass		
Glass, borosilicate	All analyte groups except silica and boron.	

Adapted from USGS Field Manual, Chapter 2, January 2000.

¹ Refers to construction material of the portions of the sampling equipment that come in contact with the sample (e.g., housing of variable speed submersible pump must be stainless steel if extractable organics are sampled; the housing of a variable speed submersible pump used to sample metals may be plastic.)

² Corroded/weathered surfaces are active sorption sites for organic compounds.

³ Plastics used in connection with inorganic trace element samples (including metals) must be uncolored or white.

⁴ May be allowable for specialized parts where no alternative material exists (e.g., Viton seals are the best available seal for some dedicated pump systems), however, contact with the sample must be minimized.

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Extractable Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Volatile Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Metals	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass (except silica and boron)
Ultratrace Metals	Teflon Polyethylene (All types) Polypropylene (All types) Polycarbonate Mercury must be in glass or Teflon
Inorganic Nonmetallics	Teflon Stainless steel Low carbon, Galvanized or Carbon steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Brass

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Microbiological samples	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Sterilize all sample containers. Thoroughly clean sampling equipment and rinse several times with sample water before collection. Sampling equipment does not require sterilization Do not rinse sample containers

**Table FS 1000-3
 Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
WATER SAMPLING					
GROUNDWATER					
1 Positive displacement pumps ²					
a. Submersible (turbine, helical rotor, gear driven)	SS, Teflon	SS, Teflon, PE, PP	Purging	All analyte groups	^{3,4,5} ; must be variable speed
			Sampling	All analyte groups	^{3,4,5} must be variable speed
	SS, Teflon	Non-inert ⁶	Purging	All analyte groups	^{3,4,5} must be variable speed; polishing required ⁷
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert ⁶	Non-inert ⁶	Purging	All analyte groups	^{3,4,5} must be variable speed; polishing required ⁷
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
b. Bladder pump (no gas contact)	SS, Teflon, PE, PP or PVC if permanently installed	SS, Teflon, PE, PP	Purging	All analyte groups	^{3,4,5} must be variable speed
			Sampling	All analyte groups	^{3,4} must be variable speed Bladder must be Teflon if sampling for volatile or extractable organics or PE or PP if used in portable pumps
	SS, Teflon, PE, PP	Non-inert ⁶	Purging	All analyte groups	^{3,4} must be variable speed; polishing required ⁷
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	This configuration is not recommended ^{3,4} must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert ⁶	Non-inert ⁶	Purging	All analyte groups	^{3,4} must be variable speed; polishing required ⁷
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	^{3,4} must be variable speed; polishing required ⁷ If sampling for metals, the tubing must be non-metallic if not SS

**Table FS 1000-3
 Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
2. Suction lift pumps					
a. Centrifugal	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	⁴ foot-valve required Must be variable speed
	N/A	Non-inert ⁶	Purging	All analyte groups	⁴ foot-valve required; polishing required Must be variable speed
b. Peristaltic	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	⁴ foot-valve required; polishing required or continuous pumping required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile organics	⁴ Silicone tubing in pump head Must be variable speed
	N/A	Non-inert ⁶	Purging	All analyte groups	⁴ foot-valve required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	⁴ Silicone tubing in pump head Must be variable speed
3. Bailers					
	SS, Teflon, PE, PP	N/A	Purging	All analyte groups	None; not recommended
		N/A	Sampling	All analyte groups	None; not recommended
	Non-inert ⁶	N/A	Purging	All analyte groups <u>except</u> volatile and extractable organics	None; not recommended If sampling for metals, the tubing must be non-metallic if not SS
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	None; not recommended If sampling for metals, the tubing must be non-metallic if not SS
<u>SURFACE WATER</u>					
1. Intermediate containers such as pond sampler, scoops, beakers, buckets, and dippers	SS, Teflon, Teflon-coated, PE, PP	N/A	Grab sampling	All analyte groups	None
	Glass	N/A		All analyte groups except boron and fluoride	None
	Non-inert ⁶	N/A		All analyte groups <u>except</u> volatile and extractable organics	None

**Table FS 1000-3
 Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
2. Nansen, Kemmerer, Van Dorn, Alpha and Beta Samplers, Niskin (or equivalent)	SS, Teflon, Teflon-coated, PE, PP	N/A	Specific depth grab sampling	All analyte groups	None
	Non-inert ^o	N/A		All analyte groups <u>except</u> volatile and extractable organics	None
3. DO Dunker	SS, Teflon, glass, PE, PP	N/A	Water column composite sampling	All analyte groups	None
4. Bailers – double valve	SS, Teflon, PE, PP	N/A	Grab sampling	All analyte groups	None
	Non-inert ^o	N/A	Grab sampling	All analyte groups <u>except</u> volatile and extractable organics	None If sampling for metals, the tubing must be non-metallic if not SS
5. Peristaltic pump	N/A	SS, Teflon, PE, PP	Specific depth sampling	All analyte groups <u>except</u> volatile organics	Silicone tubing in pump head Must be variable speed
	N/A	Non-inert ^o		All analyte groups <u>except</u> volatile and extractable organics	Silicone tubing in pump head Must be variable speed
<u>FIELD FILTRATION UNITS</u>	N/A		Dissolved constituents	Inorganic nonmetallics and metals in surface water Inorganic nonmetallics in groundwater Metals in groundwater and static wastewater and surface water Metals in moving surface water (i.e., river/stream)	Must use a 0.45 µm filter Must use a 0.45 µm filter Must use in-line, high capacity, one-piece molded filter that is connected to the outlet of a pump; no intermediate vessels; positive pressure PE, PP & Teflon bailers acceptable Must use a 1 µm filter in groundwater, a 0.45 µm filter in surface water Must use positive pressure device, but an intermediate vessel may be used. Use a 0.45 µm filter

**Table FS 1000-3
 Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
SOLID SAMPLING					
SOILS					
1. Core barrel (or liner)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	9, 10, 11
	Non-inert ⁶ nonmetallics	N/A	Sampling	All analyte groups	12
	Non-inert ⁶ metals	N/A	Sampling	All analyte groups	12
2. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups ⁸	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics must grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups <u>except</u> volatile and extractable organics	None Must be nonmetallic if not SS
3. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	11
			Compositing or homogenizing	All analyte groups except volatile organics	11
	Non-inert ⁶	N/A	Compositing or homogenizing	All analyte groups	10,11,12 must be nonmetallic if not SS
4. Shovel, bucket auger	SS	N/A	Sampling	All analyte groups ⁸	None
	Non-SS	N/A	Sampling	All analyte groups ⁸	10,11,12
5. Split spoon	SS or carbon steel w/ Teflon insert	N/A	Sampling	All analyte groups ⁸	10,11,12
6. Shelby tube	SS	N/A	Sampling	All analyte groups ⁸	9
	Carbon steel	N/A	Sampling	All analyte groups	9,10,12
SEDIMENT					
1. Coring devices	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	9,10,11

**Table FS 1000-3
Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
	Non-inert ⁶ nonmetallics	N/A	Sampling	All analyte groups	¹²
	Non-inert ⁶ metals	N/A	Sampling	All analyte groups	^{9,10,11}
2. Grab – Young, Petersen, Shipek	Teflon, Teflon-lined, SS	N/A	Sampling	All analyte groups ⁸	None
	Carbon steel	N/A	Sampling	All analyte groups	^{10,11}
3. Dredges – Eckman, Ponar, Petit Ponar Van Veen	SS	N/A	Sampling	All analyte groups ⁸	None
	Carbon steel, brass	N/A	Sampling	All analyte groups	^{10,11}
4. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon- coated, PE, PP	N/A	Sampling	All analyte groups ⁸	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics be grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups <u>except</u> volatile and extractable organics	None must be nonmetallic if not SS
5. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups ⁸	¹¹
			Compositing or homogenizing	All analyte groups except volatile organics	¹¹
	Non-inert ⁶	N/A	Compositing or homogenizing	All analyte groups <u>except</u> volatile and extractable organics	none ¹¹ must be nonmetallic if not SS
WASTE ¹³					
Scoop	SS	N/A	Liquids, solids & sludges	All analyte groups ⁸	Cannot collect deeper phases
Spoon	SS	N/A	Solids, sludges	All analyte groups ⁸	Cannot collect deeper phases
Push tube	SS	N/A	Solids, sludges	All analyte groups ⁸	Cannot collect deeper phases
Auger	SS	N/A	Solids	All analyte groups ⁸	None

**Table FS 1000-3
 Equipment Use and Construction**

<u>EQUIPMENT</u>	<u>CONSTRUCTION</u>		<u>USE</u>	<u>PERMISSIBLE ANALYTE GROUPS</u>	<u>RESTRICTIONS AND PRECAUTIONS</u>
	<u>HOUSING</u> ¹	<u>TUBING</u>			
Sediment sampler	SS	N/A	Impoundments, piles	All analyte groups ⁸	None
Ponar dredge	SS	N/A	Solids, sludges & sediments	All analyte groups ⁸	None
Coliwasa, Drum thief	Glass	N/A	Liquids, sludges	All analyte groups	None
Mucksucker, Dipstick	Teflon		Liquids, sludges	All analyte groups	Not recommended for tanks > 11 feet deep
Bacon bomb	SS	N/A	Liquids	All analyte groups ⁸	Not recommended for viscous wastes
Bailer	SS, Teflon	N/A	Liquids	All analyte groups ⁸	Do not use with heterogeneous wastes Not recommended for viscous wastes
Peristaltic pump	N/A	Teflon, Glass	Liquids	All analyte groups except volatile organics	Do not use in flammable atmosphere Not recommended for viscous wastes
Backhoe bucket	Steel	N/A	Solids, Sludges		Difficult to clean Volatiles and metals must be taken from the interior part of the sample
Split spoon	SS	N/A	Solids	All analyte groups ⁸	
Roto-Hammer	Steel	N/A	Solids	All analyte groups ⁸	Physically breaks up sample Not for flammable atmospheres

Acronyms:

N/A not applicable
 SS stainless steel
 HDPE high-density polyethylene
 PE polyethylene
 PVC polyvinyl chloride
 PP polypropylene

Table FS 1000-3
Equipment Use and Construction

- ¹ Refers to tubing and pump housings/internal parts that are in contact with purged or sampled water (interior and exterior of delivery tube, inner lining of the discharge tube, etc.).
- ² If used to collect volatile or extractable organics, all power cords and other tubing must be encased in Teflon, PE or PP.
- ³ If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.
- ⁴ Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between uses.
- ⁵ In-line check valve required.
- ⁶ "Non-inert" pertains to materials that are reactive (adsorb, absorb, etc.) to the analytes being sampled. For organics, materials include rubber, plastics (except PE and PP), and PVC. For metals, materials include brass, galvanized, and carbon steel.
- ⁷ "Polishing": When purging for volatile or extractable organics, the entire length of tubing or the portion which comes in contact with the formation water must be constructed of Teflon, SS, PE or PP. If other materials (e.g., PVC, garden hoses, etc.) are used, the following protocols must be followed: 1) slowly withdraw the pump from the water column during the last phase of purging, to remove any water from the well that may have contacted the exterior of the pump and/or tubing; 2) remove a single well volume with the sampling device before sampling begins. **Do not use Tygon** for purging if purgeable or extractable organics are of interest. Polishing **is not recommended**; use of sampling equipment constructed of appropriate materials is preferred.
- ⁸ Do not use if collecting for hexavalent chromium (Chromium⁺⁶)
- ⁹ If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.
- ¹⁰ If a non-stainless steel (carbon steel, aluminum) liner, core barrel or implement is used, take the samples for metals, purgeable organics and organics from the interior part of the core sample.
- ¹¹ Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest.
- ¹² If non-inert-liner, core barrel or implement is used, take samples from the interior part of the collected sample.
- ¹³ If disposable equipment of alternative construction materials is used, the construction material must be compatible with the chemical composition of the waste, cannot alter the characteristics of the waste sample in any way, and cannot contribute analytes of interest or any interfering components.

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ^{6, 7}
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
Table IA— Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C ⁸	36 hours
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C ⁹	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C ⁹	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C ⁹	48 hours
10. Boron	P, FP, or Quartz	HNO ₃ to pH<2	6 months
11. Bromide	P, FP, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, FP, G	Cool, ≤6 °C ⁹	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6 °C ⁹	48 hours
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C ⁹ , NaOH to pH>12 ¹⁰ , reducing agent ⁵	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
Table IB—Metals:			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C ⁹ , pH = 9.3–9.7 ¹²	28 days
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH<2	28 days

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
 Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹³	5 mL/L 12N HCl or 5 mL/L BrCl ¹³	90 days ¹³
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70– 72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO ₃ to pH<2, or at least 24 hours prior to analysis ¹⁴	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C ⁹	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C ⁹	48 hours
41. Oil and grease	G	Cool, ≤6 °C ⁹ , HCl or H ₂ SO ₄ to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C ⁹ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤6 °C ⁹	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C ⁹	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C ⁹ , H ₂ SO ₄ to pH<2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C ⁹	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C ⁹	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C ⁹	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C ⁹	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C ⁹	7 days
61. Silica	P or Quartz	Cool, ≤6 °C ⁹	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C ⁹	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C ⁹	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C ⁹ , add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes
68. Surfactants	P, FP, G	Cool, ≤6 °C ⁹	48 hours

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C ⁹	48 hours

Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ¹⁶	14 days ¹⁶
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH to 4–5 ¹⁷	14 days ¹⁷
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
7, 38. Benzidines ^{18,19}	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction ²⁰
14, 17, 48, 50–52. Phthalate esters ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	7 days until extraction, 40 days after extraction
82–84. Nitrosamines ^{18,21}	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
88–94. PCBs ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	1 year until extraction, 1 year after extraction
54, 55, 75, 79. Nitroaromatics and isophorone ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 87. Haloethers ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction, 40 days after extraction
29, 35–37, 63–65, 107. Chlorinated hydrocarbons ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹	7 days until extraction, 40 days after extraction
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs ¹⁸			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C ⁹ , 0.008% Na ₂ S ₂ O ₃ ⁵ , pH<9	1 year

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container ¹	Preservation ^{2, 3}	Maximum holding time ⁴
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C ⁹	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C ⁹	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year
Table ID—Pesticides			
Tests: 1–70. Pesticides ¹⁸	G, FP-lined cap	Cool, ≤6 °C ⁹ , pH 5–9 ²²	7 days until extraction, 40 days after extraction
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO ₃ to pH<2	6 months
Table IH—Bacterial Tests:			
1. <i>E. coli</i>			
2. Enterococci	PA, G	Cool, <10 °C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours ⁶
Table IH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours. ²³
9. Giardia	LDPE; field filtration	0–8 °C	96 hours ²³

Reference: This table is adapted from Table II, 40 CFR Part 136, 2007

¹ “P” is polyethylene; “FP” is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; “G” is glass; “PA” is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); “LDPE” is low density polyethylene.

² Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample,

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

³ When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁴ Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

⁵ Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$), ascorbic acid, sodium arsenite (NaAsO_2), or sodium borohydride (NaBH_4). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If NaBH_4 or NaAsO_2 is used, 25 mg/L NaBH_4 or 100 mg/L NaAsO_2 will reduce more than 50 mg/L of chlorine (see method “Kelada-01” and/or Standard Method

4500–CN⁻ for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

⁶ Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

⁷ For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

⁸ Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

⁹ Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “ ≤ 6 °C” is used in place of the “4 °C” and “ < 4 °C” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures ($1/100^{\text{th}}$ of 1 degree); rather, three

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

¹⁰ Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) SULFUR: To remove elemental sulfur (S₈), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) SULFIDE: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH

< 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times
Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in μg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) SULFITE, THIOSULFATE, OR THIOCYANATE: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA-1677) to preclude cyanide loss or positive interference.

(4) ALDEHYDE: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) CARBONATE: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥ 12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500-CN.B.3.d).

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

(6) CHLORINE, HYPOCHLORITE, OR OTHER OXIDANT: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

¹¹ For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

¹² To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

¹³ Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

¹⁴ An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

¹⁵ Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

¹⁶ If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

¹⁷ The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

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¹⁸ When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤ 6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 19, 20 (regarding the analysis of benzidine).

¹⁹ If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

²⁰ Extracts may be stored up to 30 days at < 0 °C.

²¹ For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling

²² The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

²³ Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

Analyte	Methods	Reference ¹	Container ²	Preservation ³	Maximum Holding Time ⁴
Bromine	DPD Colorimetric ⁵	SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 ⁶	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G ⁷	Dark 4°C Filtered, dark, 20°C	48 hours chilled until filtration ⁸ , and analyze immediately or 48 hours chilled until filtration ⁸ , and 28 days (frozen) after filtration
Corrosivity	Calculated (CaCO ₃ Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C ⁹	7 days ⁹
FL-PRO	Gas Chromatography	DEP (11/1/95)	G only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	7 days until extraction, 40 days after extraction
Odor	Human Panel	SM 2150	G only	Cool 4°C	6 hours
Salinity	Electrometric ¹⁰ Hydrometric ¹⁰	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days ¹⁰
Taste	Human Panel	SM 2160 B, C, D ASTM E679-91	G only	Cool 4°C	24 hours
Total Dissolved Gases	Direct-sensing Membrane-diffusion	SM 2810	_____	_____	Analyze in-situ
Total Petroleum Hydrocarbons	Gravimetry	EPA 1664	G only	Cool 4°C, H ₂ SO ₄ or HCl to pH<2	28 days
Transparency	Irradiometric ¹¹	62-302.200(6), FAC	_____	_____	Analyze in-situ
Un-ionized Ammonia	Calculated ¹²	DEP-SOP ¹³	P, G	Cool 4°C Na ₂ S ₂ O ₃ ¹²	8 hours unpreserved 28 days preserved ¹²
Organic Pesticides ¹⁴	GC and HPLC	EPA (600-series) ¹⁴	¹⁵	¹⁵	¹⁵

¹ SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 20th edition, 1998 and Standard Methods Online.

ASTM XXXX-YY = procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1999.

² P = plastic, G = glass.

³ When specified, sample preservation should be performed immediately upon sample collection.

⁴ The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

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- ⁵ The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).
- ⁶ The Determination of Inorganic Anions in Water by Ion Chromatography", EPA Method 300.0, Revised August 1993, by John D. Pfaff, U. S. EPA Cincinnati, Ohio 45268.
- ⁷ Collect samples in opaque bottles and process under reduced light.
- ⁸ Samples must be filtered within 48 hours of collection. Add magnesium carbonate to the filter while the last of the sample passes through the filter..
- ⁹ Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.
- ¹⁰ The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.
- ¹¹ Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DEP Chapter 62-302, FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.
- ¹² The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on-site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H₂SO₄ to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.
- ¹³ DEP Central Analytical Laboratory, Tallahassee, FL, Revision No. 2, 2-12-2001. The document is available from the DEP Standards & Assessment Section..
- ¹⁴ Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) that are not included in Table ID of 40 CFR Part 136 (July 2007).
- ¹⁵ Container, preservation and holding time as specified in each individual method must be followed.

Table FS 1000-6
Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

Analyte	Methods	References	Container	Preservation	Maximum Holding Times
Volatile Organics	Purge-and-Trap GC and GC-MS	8015, 8260, 8021, 5035	See Table 1000-7		
Semivolatile Organics	GC, HPLC, and GC-MS	8041, 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 8141, 8151, 8270, 8275, 8280, 8290, 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332, 8410, 8430, 8440, FL-PRO	Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C ¹	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C ¹ in dark	30 days until extraction, 45 days after extraction
Total Metals-except mercury and chromium VI methods	Flame AA, Furnace AA, Hydride and ICP	All 7000-series (except 7195, 7196, 7197, 7198, 7470 and 7471), and 6010 (ICP)	Glass or plastic 8 oz widemouth (200 grams sample)	None	6 months
Chromium VI	Colorimetric, Chelation with Flame AA (200 gram sample)	7196 and 7197 (prep 3060)	Glass or plastic, 8 oz widemouth (200 gram sample)	Cool 4°± 2°C ¹	1 month until extraction, 4 days after extraction ²
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4°± 2°C ¹	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool 4°C ¹	24 hours
Aggregate Properties			Glass or plastic	Cool 4°C ¹	14 days
Inorganic nonmetallics all except:			Glass or plastic		28 days
Sulfite, Nitrate,			Glass or plastic	Cool 4°C ¹	48 hours
Nitrite & o-phosphate					
Elemental Phosphorus			Glass		48 hours

Table FS 1000-6

Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

The term "residuals" include: (1) sludges of domestic origin having no specific requirements in Tables FS-1000-4 or FS-1000-9; (2) sludges of industrial origin; and (3) concentrated waste samples.

¹ Keep soils, sediments and sludges cool at 4°C from collection time until analysis. No preservation is required for concentrated waste samples.

² Storage Temperature is 4°C, ±2°C

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

Conc. Level	Sampling Device	Collection Procedure	Sample Container		Preservation	Sample Preparation	Max HT ^①	Determinative Procedure
			Type	Vial Preparation				
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1	NaHSO ₄ / 4°C	5035 - Section 7.2	14 D	Any recognized VOC Method
				5035 - 6.1.1 ^②	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
				5035 - 6.1.1 ^②	4°C / -10°C ^{③,④}	5035 - Section 7.2	48 H / 14 D ^⑤	Any recognized VOC Method
	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{②,⑥,⑦}	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 ^{⑥,⑦}	NaHSO ₄ / 4°C	5035 - Section 7.2 ^⑥	48 H / 14 D ^⑤	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1 ^{②⑥⑦}	4°C / -10°C ^{③,④}	5035 - Section 7.2 ^⑥	48 H / 14 D ^⑤	Any recognized VOC Method
>200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3 ^⑥	EnCore or equivalent	5035 - 6.1.3 ^{⑥,⑦}	4°C	5035 - Sections 7.3.2 & 7.3.3 ^⑥	48 H / 14 D ^⑤	Any recognized VOC Method
>200 ug/kg ^⑧	Coring Device	5035 - Section 6.2.2.3 ^⑧	Glass Vial w/ PTFE-silicone Septum	6.1.3 ^⑧	Methanol/PEG + 4°C	5035 - Section 7.3.4	14 D	Any recognized VOC Method
	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum	6.1.2	4°C	5035 - Sections 7.3.1 - 7.3.3	14 D	Any recognized VOC Method
Oily Waste	Conventional Devices	5035 - Section 6.2.4.2	Glass w/ PTFE-silicone Septum	6.1.4	4°C	5035 - Sections 7.4.1 - 7.4.2	14 D	Any recognized VOC Method
	Conventional Devices	5035 - Section 6.2.4.1	Glass w/ PTFE-silicone Septum	6.1.4	Methanol/PEG + 4°C	5035 - Sections 7.4.3	14 D	Any recognized VOC Method
Dry Wt.	Conventional Devices		Glass with Teflon liner		4°C	5035 - Section 7.5		
Soil Screen	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum		4°C	5035 - Section 7.1	14 D	Any recognized VOC Method

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

- ① Maximum time allowable from time/date of collection to sample analysis.
- ② Eliminate 6.1.1.2; use only organic-free water.
- ③ Contents of sampling device must be transported to the laboratory at 4°C and stored at -10°C.
- ④ In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle to maximize surface area.
- ⑤ Maximum allowable time at 4°C is 48 hours; maximum allowable time to sample analysis is 14 days (from time of sample collection).
- ⑥ Conducted in the laboratory.
- ⑦ Entire contents of sampling device are extruded into the sample analysis vial containing the appropriate solvent.
- ⑧ Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 ug/kg.
- ⑨ Methanolic preservation in the field is not recommended, but may be used if approved by an DEP program.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
MICROBIOLOGICAL-BACTERIA	Cool < 10°C, Na ₂ S ₂ O ₃ ⁵			P or G
Total Coliforms, fecal coliforms & <i>E. coli</i> in drinking water	Cool < 10°C ⁶ , Na ₂ S ₂ O ₃ ⁵	30 Hours ⁷		P or G
Total coliforms and fecal coliforms in source water Heterotrophic bacteria in drinking water	Cool < 10°C, Na ₂ S ₂ O ₃ ⁵	8 hours		P or G
Gross Alpha	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Gross beta	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Strontium-89	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Strontium-90	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Radium-226	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Radium-228	Conc. HCl or HNO to pH <2 ^{8,9}	6 mo		P or G
Cesium-134	Concentrated HCl to pH <<2 ^{8,9}	6 mo		P or G
Iodine-131	None	8 da		P or G
Tritium	None	6 months		G
Uranium	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Photon emitters	Conc. HCl or HNO ₃ to pH <2 ^{8,9}	6 mo		P or G
Asbestos	Cool 4°C	48 hours		P or G
Bromate	Ethylenediamine (50mg/L)	28 days		P or G
Cyanide	Cool, 4C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		P or G
Nitrate	Cool, 4°C	48 hours		P or G
Nitrate (chlorinated source)	Cool, 4°C	14 days		P or G
Odor	Cool 4°C	24 hours		G
502.2	Sodium Thiosulfate or Ascorbic Acid, 4°C HCl pH<2 if Ascorbic Acid is used	14 days		Glass with PTFE Lined Septum

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
504.1	Sodium Thiosulfate Cool, 4°C,	14 days	4°C, 24 hours	Glass with PFTE-Lined Septum
505	Sodium Thiosulfate Cool, 4°C	14 days (7 days for Heptachlor)	4°C, 24 hours	Glass with PFTE-Lined Septum
506	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
507	Sodium Thiosulfate Cool, 4°C, Dark	14 days (see method for exceptions)	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
508	Sodium Thiosulfate Cool, 4°C, Dark	7 days (see method for exceptions)	4°C, dark, 14 days	Glass with PFTE-lined Cap
508A	Cool, 4°C	14 days	30 days	Glass with PFTE-lined Cap
508.1	Sodium Sulfite, HCl pH<2, Cool, 4°C	14 days (see method for exceptions)	30 days	Glass with PFTE-lined Cap
515.1	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 28 days	Amber Glass with PFTE-lined Cap
515.2	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.3	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.4	Sodium Sulfite, HCl pH<2, Cool, ≤10°C for first 48 hours ≤6°C thereafter, Dark	14 days	≤0°C, 21 days	
524.2	Ascorbic Acid, HCl pH<2, Cool 4°C	14 days		Glass with PFTE-lined Septum
525.2	Sodium Sulfite, Dark, Cool, 4°C, HCl pH<2	14 days (see method for exceptions)	≤ 4°C, 30 days from collection	Amber Glass with PFTE-lined Cap
531.1, 6610	Sodium Thiosulfate Monochloroacetic acid, pH<3, Cool, 4°C	Cool 4°C, 28 days		Glass with PFTE-lined Septum
531.2	Sodium Thiosulfate, Potassium Dihydrogen Citrate buffer to pH 4,	28 days		

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation ¹	Holding Time ²	Holding Time for Extract ³	Container ⁴
	dark, ≤10°C for first 48 hr, ≤6°C thereafter			
547	Sodium Thiosulfate Cool, 4°C	14 days (18 mo. frozen)		Glass with PTFE-lined Septum
548.1	Sodium Thiosulfate (HCl pH 1.5-2 if high biological activity), Cool, 4°C, Dark	7 days	≤4°C 14 days	Amber Glass with PTFE-lined Septum
549.2	Sodium Thiosulfate (H ₂ SO ₄ pH<2 if biologically active), Cool, 4°C, Dark	7 days	21 days	High Density Amber Plastic or Silanized Amber Glass
550, 550.1	Sodium Thiosulfate Cool, 4°C, HCl pH<2	7 days	550, 30 days 550.1, 40 days Dark, 4°C	Amber Glass with PTFE-lined Cap
551.1	Sodium Thiosulfate, Sodium Sulfite, Ammonium Chloride, pH 4.5-5.0 with phosphate buffer, Cool, 4°C	14 days		Glass with PTFE-lined Septum
552.1	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 48 hours	Amber Glass with PTFE-lined cap
552.2	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 7 days ≤-10°C 14 days	Amber Glass with PTFE-lined cap
555	Sodium Sulfite, HCl, pH ≤ 2, Dark, Cool 4°C	14 days		Glass with PTFE-lined cap
1613B	Sodium Thiosulfate, Cool, 0-4°C, Dark		Recommend 40 days	Amber Glass with PTFE-lined Cap

¹ Preservation, when required, must be done immediately upon sample collection.

² Stated values are the maximum regulatory holding times. Sample processing must begin by the stated time.

³ Stated time is the maximum time a prepared sample extract may be held before analysis.

⁴ (P) polyethylene or (G) or glass. For microbiology, plastic sample containers must be made of sterilizable materials (poly-propylene or other autoclavable plastic).

⁵ Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.

FS 1000-8

Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

- ⁶ Temperature requirement applies only to source water samples, however once received by the laboratory, if sample processing does not begin on the same working day, samples must be refrigerated.
- ⁷ If samples are analyzed after 30 hours, but within 48 hours of collection, the laboratory is to indicate in the analytical report that the data may be invalid because of excessive delay in sample processing. No samples received after 48 hours are to be accepted or analyzed for compliance with the regulations of the Department of Environmental Protection or the Department of Health.
- ⁸ It is recommended that the preservative be added at the time of collection unless suspended solids activity is to be measured. It is also recommended that samples be filtered, if suspended or settleable solids are present, prior to adding preservative, at the time of collection. However, if the sample has to be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.
- ⁹ If HCl is used to acidify samples, which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

Table FS 1000-9
Containers, Preservation and Holding Times for Biosolids Samples and Protozoans

<i>ANALYTE NAME</i>	<i>CONTAINER</i>	<i>PRESERVATION</i>	<i>MAX HOLDING TIME</i>
Fecal Coliform	Plastic or Glass	Cool 4°C	24 hours
Salmonella	Plastic or Glass	< 10°C	24 hours
Enteric Viruses	Plastic or Glass	Up to 25°C	2 hours
Enteric Viruses	Plastic or Glass	2 to 10°C	48 hours
Specific Oxygen Uptake Rate	Plastic or Glass	None	As Soon As Possible
Helminth OVA	Plastic or Glass	< 4°C (Do not Freeze)	24 hours
Cryptosporidium/Giardia	Plastic or Glass	0 - 8°C (Do not Freeze)*	96 Hours
Total Solids	Plastic or Glass	≤6°C (Do not Freeze)	7 days
Metallics	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	
Other Inorganic Pollutants	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	

***Dechlorinate bulk samples when applicable**

Table FS 1000-10
Container Materials, Preservation, and Holding Times for Fish and Shellfish

Analyte	Matrix	Sample Container	Field (Transport to Lab)		Laboratory	
			Preservation	Maximum Shipping Time	Storage	Holding Time
	Whole Organism (Fish, shellfish, etc.)	Foil-wrap each organism (or composite for shellfish) and transport in waterproof plastic bag	Cool in wet ice or: ----- Freeze on dry ice	24 hours ----- 48 hours		
Mercury	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	28 days
Other metals	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	6 months
Organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	1 year
Dioxin	Tissue (fillets and edible portions, homogenates)	Amber containers: Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	30 days until extraction, 15 days after extraction
Lipids	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	1 year

PTFE = Polytetrafluoroethylene (Teflon)

Table FS 1000-11
Holding Times for SPLP or TCLP Extraction, Sample Preparation and Determinative Analysis

Holding Time (Days)				
	From: Field Collection	From: SPLP or TCLP Extraction	From: Preparative Extraction	Total Elapsed Time
	To: SPLP or TCLP Extraction	To: Preparative Extraction	To: Determinative Analysis	
Volatiles	14	NA	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Mercury	180	NA	180	360

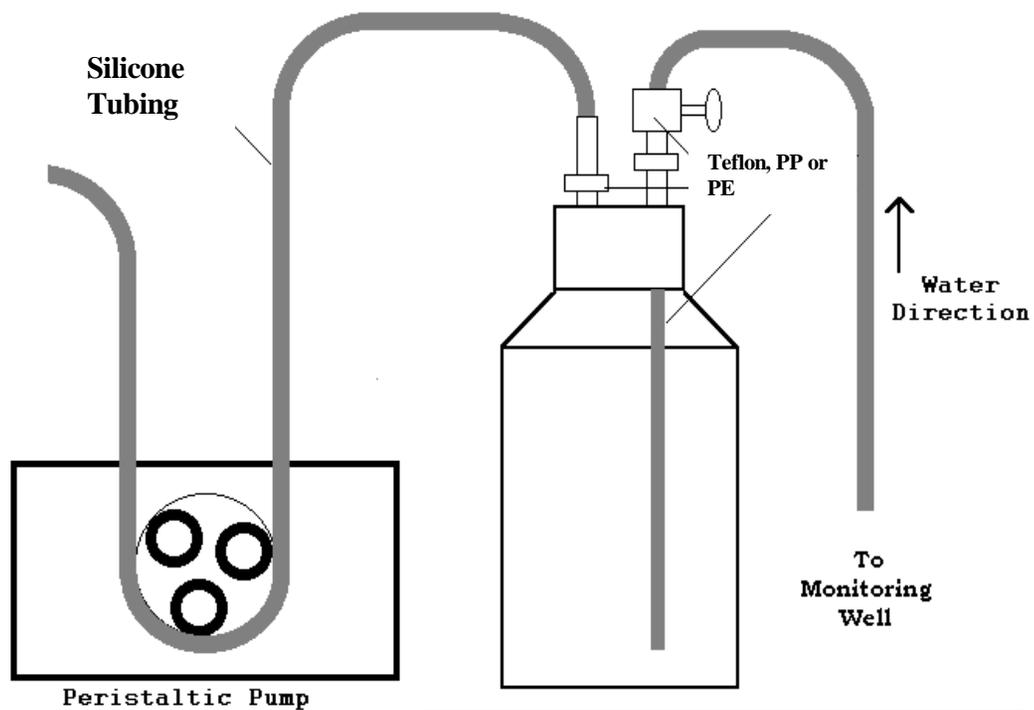
NA – Not Applicable

**Table FS 1000-12
 Preventive Maintenance Tasks**

INSTRUMENT/ACTIVITY	FREQUENCY
REFRIGERATORS, INCUBATORS, OVENS	
Clean interior	Monthly
Check thermometer temperature against certified thermometer or equivalent	Annually
ANYTICAL BALANCES	
Clean pan and compartment	Daily ¹
Check with Class S weights	Monthly
Manufacturer cleaning and calibration	Annually
pH AND ION SELECTIVE ELECTRODES	
PROBE	
Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	Daily, Replace as necessary
Check response time	Daily ¹
METER	
Check batteries and electronics for loose connections and cracked leads	Daily ¹ , Replace as necessary
TURBIDIMETER	
Clean instrument housing	Monthly
Clean cells	Daily ¹
CONDUCTIVITY METER	
Check batteries and probe cables	Daily ¹
Replatinize Probe	Per manufacturer's recommendations
DISSOLVED OXYGEN METERS	
PROBE	
Check membrane for deterioration; check filling solution	Daily ¹ , Replace as necessary
METER	
Battery level and electronics checked	Daily ¹ , Replace as necessary
THERMOMETERS	
Check for cracks and gaps in the mercury	Daily ¹ , Replace as necessary
TEMPERATURE PROBE	
Check connections, cables	Daily ¹
Check against calibrated thermometer	Daily ¹
AUTOMATIC SAMPLE COLLECTION SYSTEMS (e.g., ISCO, Sigma)	
Check sampler operation (forward, reverse, automatic through three cycles of the purge-pump-purge cycle)	Daily ¹ Prior to Sampling Event
Check purge-pump-purge cycle when sampler is installed	Daily ¹ Prior to Sampling Event
Check the flow pacer that activates the sampler to assure proper operation	Daily ¹ Prior to Sampling Event
Check desiccant	Daily ¹ , Replace as Necessary
Check batteries	Daily ¹ , Replace as Necessary
Check pumping rate against manufacturer's specifications	Daily ¹ , Replace as Necessary

¹Daily is defined as prior to use or a 12-hour period if equipment is run continuously

Figure FS 1000-1
Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump



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.

FS 2000. GENERAL AQUEOUS SAMPLING

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

1. COMMON PROCEDURES

The following procedures are applicable to the collection of all water samples.

1.1. Refer to FS 1000 for procedures that are common to all types of sample collection including general preservation and thermal preservation procedures.

1.2. Grab Samples

1.2.1.1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15-minute time limit applies to aqueous samples only. No time limit applies to the collection of solid samples (e.g., residuals).

1.2.1.2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- A snapshot of the water quality at a particular instant in time is desired.
- The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow).
- The characteristics of the water or waste stream are known to be constant or nearly so.
- When conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- The sample is to be analyzed for analytes whose characteristics are likely to change significantly with time (e.g., dissolved gases, microbiological tests, pH).
- The sample is to be collected for analytes such as Oil and Grease, bacteriological tests or other parameters listed in number 3 of this section where the compositing process could significantly affect the actual concentration.
- Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.
- When identifying and tracking slug loads and spills.

1.2.1.3. If required, measure the following parameters on grab samples or in-situ.

NOTE: If the permit specifies a composite sample for any of the parameters mentioned below, **FOLLOW THE PERMIT CONDITIONS**

Cyanide	Oil and Grease
Residual Chlorine	pH
Dissolved constituents in field-filtered samples (ortho-phosphorus, metals, etc.)	Specific Conductance
Dissolved Oxygen and other dissolved gases	Un-ionized Ammonia
Microbiological Parameters	Volatile Organic Compounds
TRPHs	Temperature
Total Phenols	

1.3. Composite Samples

1.3.1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.

1.3.2. Composite samples are used when stipulated in a permit or when:

- The water or wastewater stream is continuous;
- Analytical capabilities are limited;
- Determining average pollutant concentration during the compositing period;
- Calculating mass/unit time loadings; or
- Associating average flow data to parameter concentrations

1.3.3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow rate or they may be collected proportional to the flow rate. The permit or work plan will specify which composite sample type to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

1.3.3.1. Time Composite Sample: Time composite samples are based on a constant time interval between samples. A time composite sample can be collected manually or with an automatic sampler. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample described in number 3.3 of this section.

1.3.3.2. Flow Proportional Composite Sample: Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. There are two methods used to collect this type of sample:

- Method 1: Collect a constant sample volume per stream flow (e.g., a 200 mL sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately.
- Method 2: Collect a sample by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

1.3.3.3. Sequential Composite Sample: Sequential composite samples are composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited for each hour.

- The 24-hour composite is made up from the individual one-hour composites. Each of the 24 individual samples is manually flow-proportioned according to the flow recorded for the hour that the sample represents. Each flow-proportioned sample is then added to the composite samples. The actual compositing of the samples is done by hand and may be done in the field or the laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to, and handled, in the laboratory. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.
- A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow
- Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period. For example, for pretreatment studies you can visually determine when high strength wastes are being discharged from a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure the pH throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals

1.3.3.4. Continuous Composite Sample: Collected continuously from the stream. The sample may be a constant volume that is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

1.3.3.5. Areal Composite: A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites must be made up of equal volumes of grab samples; each grab sample must be collected in an identical manner. Examples include residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc sample is similar to the flow proportional composite.

1.4. Collection Techniques

1.4.1. When filling a sample container that already contains premeasured preservative, slowly pour the sample down the side of the container so that the preservative does not

splatter. If the preservative is concentrated acid, and the sample water is added too quickly, the reaction between the water and the acid can generate enough heat to burn unprotected skin or could splatter and cause acid burning.

1.4.2. Collect grab samples (single, discrete samples) unless directed by permit, program, or approved sampling plan or work plan to collect composite samples.

1.4.3. Except for volatile organic compounds and sulfide, leave ample headspace in the sample bottle to allow for expansion, effervescence and proper mixing at the laboratory.

1.5. Collecting Filtered/Dissolved Samples

1.5.1. Certain studies or projects require collection of dissolved (i.e., filtered) samples. Identify all analytes in samples that are filtered as “dissolved” or “filtered” in field notes or laboratory transmittal forms and on final reports.

1.5.2. Collect both filtered and unfiltered samples from the same water in a collection device (e.g., bailer, intermediate container) or consecutively if sampling from a pump.

1.5.3. Collect dissolved metals in groundwater according to the procedures discussed in FS 2225. **Do not** collect filtered samples for metals from groundwater sources unless:

1.5.3.1. The DEP has required or approved the protocol and the DEP program allows the use of the procedure; or

1.5.3.2. The organization is documenting that a filtered groundwater sample is as or more representative of the groundwater quality. In this case, collect **both** unfiltered and filtered samples for analysis. Submit the results of both samples the DEP for review.

1.5.4. Filtration, when performed, must begin within 15 minutes of sample collection.

1.5.5. Collect dissolved groundwater samples for metals with a one-piece molded construction 1 µm filter unless otherwise specified by a DEP program. Use a 0.45 µm filter when filtering all other constituents **including** metals in surface water.

1.5.6. The filter must be compatible with the analyte to be filtered (e.g., zero carbon content for carbon analysis; non-protein binding filters for nitrogen).

1.5.7. Equipment blanks, when collected, must be processed through the filtration apparatus and analyzed for the analytes of interest.

1.5.8. Filters and filtration equipment are intermediate devices and therefore must be adequately rinsed per FS 2110 section 1.1.2.1.

THE FOLLOWING ARE SPECIAL CONSIDERATIONS FOR VARIOUS ANALYTE GROUPS:

FS 2001. *pH-Preserved Samples*

1. SAMPLE CONTAINERS

1.1. Use properly cleaned sample containers (see FC 1300).

1.2. Inspect all containers for visual defects or contamination. Discard if defects are present or containers do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. If the preservative is added after the sample is collected, (the container is not prepreserved), do not fill the container to the rim.

3. PRESERVATION

3.1. Preserve the sample within 15 minutes of sample collection or filtration (if applicable) unless collected as a composite sample (see FS 1006, section 1.3) or for analysis of lead and copper for drinking water compliance (see FS 2310, section 2).

3.2. Preserve the sample with the chemical specified by the method or preservation tables (Tables FS 1000-4 to FS 1000-10).

3.2.1. The chemical reagents must be pure enough so that the reagent does not contribute contamination or interferences to the analytes of interest.

3.3. Preserve the sample by adding an accurately measured amount of preservative to the container. Premeasured vials of the preservative, or a graduated container or pipet, may be used.

3.3.1. Tightly cap the sample container and gently tip the container two to three times to distribute the chemical.

3.4. The pH of the preserved sample must meet the pH criterion of the applicable preservation tables (see Tables FS 1000-4 to FS 1000-10). **Do not over preserve the sample.**

3.4.1. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH meets the required level. **Do not put the pH paper directly into the sample container.**

3.4.2. If the pH does not meet the required level, add additional measured amounts of preservative and test with narrow range pH paper (see section 3.4.1 above) until the pH meets the pH requirement.

3.4.3. Record the total amount of preservative that was added to the sample. This documentation is necessary for the next site visit, since additional acid may be needed to adequately preserve the sample on subsequent visits.

3.5. Cooling to less than 6°C with wet ice (see FS 1006, section 5) may be required.

3.6. Protect from direct sunlight.

3.7. Preserve at least one of the equipment blanks with the **greatest** amount of preservative that was required in the sample set and note the amount in field documentation.

3.8. After the sample has been preserved, screw the cap on tightly.

4. Verifying pH-Preserved Samples: Verify the pH of all pH-preserved samples (except volatile organics) in the field (see FS 2001, section 3.4). If samples are routinely collected from the same sample location, a pH check is not required each time samples are collected.

4.1. If the frequency of sample collection at a specified location is once per month or greater (e.g., weekly or daily), check the pH of **at least one** sample per parameter group according to the following schedule:

- 4.1.1. Weekly sampling: 1 pH check per month
 - 4.1.2. Daily sampling: 1 pH check per week
 - 4.2. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.
 - 4.2.1. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.
 - 4.2.2. For all other sample collection frequencies, pH checks may be reduced as follows:
 - 4.2.2.1. During the first sampling event at a particular site, check **all** samples (except volatile organics) that are pH-adjusted, and
 - 4.2.2.2. During subsequent visits to a particular site, check **at least one** sample per parameter group that must be pH-adjusted.
5. DOCUMENTATION
 - 5.1. Complete the sample container label and stick firmly on the container.
 - 5.2. Complete the field notes.
 - 5.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment or preservation problems.

FS 2002. *Metals*

1. SAMPLE CONTAINERS
 - 1.1. Use properly cleaned containers (see FC 1300).
 - 1.2. Inspect the containers and caps for visual defects or contamination. Do not use containers if defects are present or if they do not appear clean.
2. SAMPLE COLLECTION PROCEDURES
 - 2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.
 - 2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.
3. PRESERVATION - Follow preservation procedures outlined in FS 2001 above.
 - 3.1. Requirements for specific metals:
 - 3.1.1. For boron or cold-vapor atomic absorption Mercury with a grade of nitric acid (HNO₃) that is suitable for use for metals analysis. Use concentrated HNO₃ or 1:1 HNO₃ to lower the pH of less than 2 S.U., but greater than 1.62 S.U.
 - 3.1.2. For Chromium VI add sufficient ammonium sulfate buffer solution specified per Table FS 1000-4 to the sample to raise the pH of the sample to a pH of 9.3 – 9.7 and place in ice (see FS 2002).
 - 3.1.3. Trace Level Mercury
 - 3.1.3.1. Collect samples for trace level mercury (<100 ug/L) in tightly-capped fluoropolymer or glass bottles.

3.1.3.2. If the samples cannot be received by the laboratory within 48 hours of sample collection, preserve the sample with BrCl or HCl solution.

3.1.3.3. For dissolved trace level mercury, samples must be filtered through a 0.45 µm filter within 24 hours of sample collection. If the samples cannot be transported to the laboratory within 24 hours, follow the procedures in FS 8200 for field filtration.

3.1.4. Samples collected for lead and copper for drinking water compliance and metals other than those listed above do not require immediate acid preservation.

3.1.4.1. When samples are not acidified with acid, the transmittal form to the laboratory must:

- Clearly state that the samples are unpreserved; and
- Request that the laboratory preserve the samples.

3.1.4.2. If samples are acidified, use concentrated HNO₃ or 1:1 HNO₃ to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.2. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. On the transmittal form, clearly identify samples that must be acidified by the laboratory (FS 2002, 3.1.3 or 3.1.4 above).

FS 2003. *Extractable Organics*

1. SAMPLE CONTAINERS

1.1. Most samples are collected in glass containers with Teflon-lined caps. Note: Teflon containers are also acceptable. There are some exceptions such as collecting samples in amber glass (e.g., nitroamines, nitroaromatics, etc.). If in doubt, verify the proper container type in Tables FS 1000-4 through FS 1000-10.

1.2. Inspect glass bottles to assure that there are no visual glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.

1.3. Collect composite samples from automatic sample collection devices in refrigerated glass or Teflon containers through Teflon, polyethylene or polypropylene tubing.

2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container without touching the interior Teflon liner.

2.2. Carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. Fill bottle with sample to almost full capacity.

3. PRESERVATION

- 3.1. In general, these types of samples must be preserved by cooling to 4°C.
 - 3.1.1. Some analyte groups require a chemical preservation. See Tables FS 1000-4 through FS 1000-10 for any additional preservation.
 - 3.1.2. If the samples for pesticides cannot be extracted within 72 hours of collection, the sample pH must be in the range of 5 to 9. If needed, adjust sample to the specified pH range with sodium hydroxide or sulfuric acid.
 - 3.1.3. Add sodium thiosulfate if residual chlorine is present.
- 3.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).
4. DOCUMENTATION
 - 4.1. Complete the sample container label and stick firmly on the container.
 - 4.2. Document when samples were placed in wet ice immediately (see FS 1006, section 5).
 - 4.3. Complete the field notes.
 - 4.4. Make notes on the lab transmittal form and the field records about any sample that appears highly contaminated or exhibits other abnormal characteristics (i.e., foaming, odor, etc.).

FS 2004. *Volatile Organics*

1. SAMPLE CONTAINERS
 - 1.1. Use a screw cap glass sample vial that is sealed with a Teflon-coated septum.
 - 1.2. Collect **at least two** vials of each sample. Some laboratories may require three or more vials, therefore verify the laboratory's policy on the number of vials they require unless the laboratory provides the sampling kit.
 - 1.3. Inspect the vials for glass or septum defects (e.g., rim must not have nicks or visible depressions and the septum must not be deformed). Do not use containers if defects are present or if they do not appear clean.
2. SAMPLE COLLECTION PROCEDURES
 - 2.1. Special precautions for petroleum sources:
 - 2.1.1. If possible, transport and store fuels in a separate vehicle from sampling equipment, empty vials and collected samples. If these items must be transported in the same vehicle as fuel, store the fuels as far away from the vials as possible.
 - 2.1.2. Place all fuel or exhaust sources downwind of the sampling location.
 - 2.1.3. Position all petroleum-fueled engines (including the vehicle) downwind of the sampling operations.
 - 2.2. Do not allow the sampling equipment or hands to touch the rim of the sample container.
 - 2.3. Do not remove septum caps from VOC vials until just prior to filling. Cap vials immediately after filling with sample.
 - 2.4. **DO NOT PRERINSE VOC VIALS.**

- 2.5. Do not aerate the sample during sample collection. If collecting from a spigot or pump, reduce the flow rate to less than 100 mL/min.
- 2.6. If preservation is required, proceed to section 3 below unless the laboratory supplied vials with premeasured quantities of acid, and the sample does not need to be dechlorinated (see 3.2 below).
- 2.6.1. If no preservation is required or if the vials are prepreserved (see 2.5 above), slowly and carefully allow the sample to flow down the **side** of the vial to minimize turbulence. Fill the vial until the surface tension holds the water in a “convex meniscus”.
- 2.6.2. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.
- 2.6.3. If using a bailer, the bailer must be equipped with a controlled flow bottom assembly.

3. PRESERVATION

- 3.1. Preserve the sample **during** the sample collection process.
- 3.2. Dechlorination: Some treated water samples (drinking water and treated wastewater) may contain residual chlorine that must be removed with a dechlorination agent such as sodium thiosulfate or ascorbic acid. This process must occur **before** any additional preservatives (i.e., acid) are added. The dechlorination agent must be **in the vial** before the sample is added.
- 3.2.1. Laboratories may supply vials with premeasured quantities of dechlorination agent. If acid preservation **is not required**, fill the vials (see section 2.5.1 above) and proceed to section 4 below.
- 3.2.2. For chlorinated drinking water samples, add 3 mg sodium thiosulfate per 40 mL vial.
- 3.2.3. If the chlorine level is unknown, the concentration must be measured (see FT 2000). For sources other than drinking water (e.g., chlorinated effluent), 10 mg sodium thiosulfate per 40 mL vial will remove up to 5 ppm Cl₂.

3.3. Acid Preservation

3.3.1. Chlorinated Samples

- 3.3.1.1. If acid preservation is required, carefully fill the vial with sample, but not to a convex meniscus as described in section 2.5.1 above.
- 3.3.1.2. Add four drops of concentrated HCl (more acid may be needed if the sample is known to contain high levels of bicarbonate or is otherwise buffered).
- 3.3.1.3. Add additional sample to create a convex meniscus.

<p>NOTE: If the sample reacts with the acid by generating gas, do not submit preserved samples for analysis. Instead, collect unpreserved samples (seven-day holding time must be met).</p>

3.3.2. Unchlorinated Samples

- 3.3.2.1. The laboratory may supply vials with premeasured quantities of acid. In this case, proceed to section 2.5.1 above. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

3.3.2.2. If the samples are preserved in the field, follow the procedure in section 3.3 above.

4. CAPPING THE VIAL

4.1. Fill the vial so that the sample surface is above the container rim (convex meniscus).

4.1.1. **Do not pour** sample into cap.

4.1.2. Fill vial from the original source (tubing, spigot, etc.) **Do not fill vial from sample collected in the cap.**

4.2. **Immediately** cap the vial with the Teflon seal contacting the sample. Some sample may overflow while tightening the cap.

4.3. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.

4.4. Turn the vial over and tap it to check for the presence of bubbles.

4.4.1. If bubbles are present, and the total volume of the bubbles is less than 5 mm in diameter, the sample may be submitted.

4.4.2. If the total volume of the bubbles is greater than 5 mm in diameter, discard the vial and fill a new one.

4.4.3. **Do not open a vial to add additional sample.**

5. SAMPLE PACKING

5.1. Label each vial with an appropriate field ID number and preservation (e.g., preserved with acid, sodium thiosulfate/acid, etc.).

5.2. Wrap each vial in a protective material (e.g., bubble wrap).

5.3. Place the set of vials in a small, sealable, untreated plastic bag unless the laboratory supplies an alternate method of packing.

5.4. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.5. Protect samples from environmental contamination during storage and transport to the laboratory.

5.6. As an added measure, DEP recommends wrapping the set of replicate samples in bubble wrap and sealing them in a container. This procedure will add further protection from potential contamination.

6. DOCUMENTATION

6.1. Label all the vials.

6.2. Complete field records.

6.3. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.

FS 2005. *Bacteriological Sampling*

1. SAMPLE CONTAINERS

1.1. Collect the samples in properly sterilized containers.

- 1.1.1. Presterilized Whirl-pak bags (or equivalent) are generally used.
- 1.1.2. If Whirl-pak bags are not used, the sample container must have a volume of at least 125 mL.
- 1.1.3. If using bottles, the caps must be sterilized. If the caps are lined, there must be documentation to show that the liner does not produce toxic compounds when sterilized.
- 1.1.4. Bottles and caps must be sterilized according to procedures in FC 1320 or purchased presterilized from a commercial vendor.

2. SAMPLE COLLECTION PROCEDURES

- 2.1. Unless a composite is specified by permit, all samples must be grab samples.
- 2.2. Do not open the container once it has been sealed.
- 2.3. Do not rinse sample container before collecting the sample.
- 2.4. Use aseptic techniques to collect the sample:
 - 2.4.1. If an intermediate device is used, thoroughly rinse with sample water. To ensure proper rinsing, DEP recommends that microbiological samples be the last sample collected with the sampling device.
 - 2.4.2. Do not put fingers into the mouth of the container or on the interior of the cap.
 - 2.4.3. Do not disinfect the sample equipment or sampling port.
- 2.5. Rinse the sampling equipment with sample water before collecting the sample. Therefore, collect microbiological samples at the end of a sampling sequence.
- 2.6. Wells with In-Place Plumbing, Spigots and/or Faucets
 - 2.6.1. Do not disinfect the spigot with bleach, alcohol or heat. Turn on spigot and flush at maximum velocity (see FS 2310).
 - 2.6.2. After flushing, reduce the water flow to approximately 500 mL/min and allow the water to flow for a few minutes before collecting samples. If other samples (metals, nutrients, etc.) are to be collected, collect these samples first.
 - 2.6.3. **Do not stop the flow before or during the filling process.**
- 2.7. Direct Grab Sample Collection
 - 2.7.1. Hold a rigid container near the base and plunge neck downward, below the surface. Turn container until the neck points slightly upward with the mouth directed toward the current. Fill to within about 1/2 inch of the top and cap immediately.
 - 2.7.2. Whirl-pak bags (or equivalent)
 - Open the bag by zipping off the top and pulling the white tabs to open the bag. Hold the bag behind the wire ties, and plunge neck downward and up in one sweeping arc; or
 - Zip off the top of the bag. Hold bag so that the mouth and wire ties are in front of the hands and fingers. Immerse the bag, and open the bag into the current.
 - The above procedures may also be accomplished by attaching the bag to a pole.
 - 2.7.2.1. Bring the bag to the surface, and press out excess water.

2.7.2.2. Seal the bag by folding the open ends at least three times and securely twisting the wire ties.

2.8. Intermediate Device Collection

2.8.1. When using an intermediate sampling device (bailer, DO dunker, niskin bottle, etc.), obtain sufficient sample in the sample collection device to completely fill the sample container. Begin pouring sample out of the device BEFORE collecting into the container. Continue to pour sample out of the device, place container under flowing stream, and fill. **Do not stop the flow before or during the filling process.**

3. PRESERVATION

3.1. Preserve samples according to Tables FS 1000-4 through FS 1000-10.

3.2. Place all samples in wet ice immediately after sample collection (see FS 1006, section 5).

3.3. When the sample contains residual chlorine, add a dechlorinating agent such as sodium thiosulfate to the sample container.

3.3.1. The final concentration of sodium thiosulfate must be approximately 100 milligrams per liter (mg/L) in the sample (add 0.1 mL of a 10% solution of thiosulfate to a 125 mL sample).

3.3.2. Some vendors or laboratories provide sterile containers with premeasured amounts of dechlorinating agent. Determine if the source of the field containers already contain a dechlorinating agent.

3.3.3. **Do not use containers with dechlorinating chemicals** when collecting samples from sources that are known to be free from residual chlorine.

4. HOLDING TIME

4.1. The holding time for microbiological samples is very short. Let the laboratory know the approximate time that samples will be collected and when they are expected to be delivered to the laboratory.

4.2. The holding time begins at the time (hours and minutes) the sample is collected and ends at the time that the sample is placed on the applicable growth media.

4.3. Consult Tables FS 1000-4, -6, -8, and -9 for holding times.

5. DOCUMENTATION

5.1. Label each sample container with an appropriate field ID number.

5.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.3. Complete field records.

5.4. Make note in the field records of any unusual sample appearances or sampling conditions.

FS 2006. *Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPHs)*

1. SAMPLE CONTAINERS

1.1. Collect samples for O&G and TRPHs in 1-liter wide mouth amber glass bottles.

- 1.2. The cap must have a Teflon liner.
- 1.3. Visually inspect glass bottles and caps for defects. Do not use container if defects are present or if they do not appear clean.
2. SELECTION OF SAMPLING POINTS
 - 2.1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.
 - 2.1.1. Select a point of greatest mixing.
 - 2.1.2. For compliance samples at a facility, collect samples from a point that best represents oil and grease concentrations.
3. SAMPLE COLLECTION PROCEDURES
 - 3.1. All samples must be grab samples.
 - 3.1.1. If composite data are required, collect individual grab samples over the specified time period.
 - 3.1.2. Submit all samples for analysis.
 - 3.1.3. Average the concentrations of the results to determine the average concentration over time.
 - 3.2. Do not collect the sample by skimming the surface.
 - 3.3. Collect a discrete sample that will be used for analysis. Do not use this sample for any other test.
 - 3.4. Remove the cap from the glass bottle without touching the interior of the container or lid.
 - 3.5. Do not rinse the sampling device or the sample container with sample water.
 - 3.6. Collect the sample directly into the container.
 - 3.6.1. If intermediate sampling equipment is needed, do not allow the sampling equipment to touch the rim of the sample container.
 - 3.6.2. Do not use automatic samplers to collect these types of samples.
 - 3.6.3. Fill the bottle with the sample water to almost full capacity.
 - 3.6.4. Add preservatives (see section 4 below).
 - 3.6.5. Quickly cap the container and tighten securely.
4. PRESERVATION
 - 4.1. Preserve the sample within 15 minutes of sample collection.
 - 4.2. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**
 - 4.3. Preserve the sample by adding an accurately measured amount of sulfuric or hydrochloric acid to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.
 - 4.3.1. Tightly cap the sample container and shake to distribute the acid.

4.3.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

4.3.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 4.3.2 above) until the pH has been reduced to below 2 pH units.

4.3.4. Record the total amount of acid that was added to the sample.

4.4. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

4.5. After the sample has been preserved, screw the cap on tightly.

4.6. Immediately place the sample in **wet** ice after preserving with acid (see FS 1006, section 5).

5. DOCUMENTATION

5.1. Label each vial with an appropriate field ID number.

5.2. Protect glass container from breakage ("bubble wrap" is recommended).

5.3. Complete field records.

5.4. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2007. *Radiological Sampling (Excludes Radon)*

1. SAMPLE CONTAINERS

1.1. Use polyethylene, polyvinyl chloride (PVC), or Teflon containers.

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

2. SAMPLE COLLECTION PROCEDURES

2.1. On unknown sites, survey the area with a beta-gamma survey instrument, such as a Geiger-Müller meter.

2.1.1. If radiation levels are above instrument background, consult a radiation safety specialist to determine appropriate safety procedures.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION

3.1. Preserve the sample with a suitable grade of nitric acid (HNO₃).

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

3.4. If the preservative is added after the sample is collected (the container is not prepreserved), do not fill the container to the rim.

3.5. Preserve the sample by adding an accurately measured volume of concentrated HNO₃ or 1:1 HNO₃ to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

3.5.1. Tightly cap the sample container and shake to distribute the acid.

3.5.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

3.5.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 3.5.2 above) until the pH has been reduced to just below 2 pH units.

3.5.4. Record the total amount of acid that was added to the sample.

3.5.5. Cooling to 4°C is not required.

3.6. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

3.7. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2008. *Radon Sampling*

Radon is a gas and is easily removed from water sources. Therefore, follow the same precautions and care used to collect volatile organic samples. Minimize contact with air during sample collection. Other sample collection techniques may be appropriate, depending on the analytical method or as specified in the project data quality objectives.

1. SAMPLE CONTAINERS

1.1. Use glass sample vials containing a premeasured portion of the scintillation "cocktail."

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

1.3. Collect at least two samples.

2. PRESERVATION: The scintillation cocktail is the only required preservative.

3. SAMPLE COLLECTION PROCEDURES Obtain specific sample collection instructions from the laboratory that will analyze the samples. These instructions must include proper handling as well as sample size and packing instructions. The following are general instructions for collecting the samples:

3.1. Carefully fill a syringe (usually 10 mL) with sample water so that air bubbles are not pulled in with the sample before, during or after filling.

3.2. Place the tip of the syringe **BELOW** the scintillation cocktail and slowly dispense the sample **BENEATH** the cocktail surface.

- 3.3. Replace the lid and cap tightly.
 - 3.4. Generally, the vial is used in the laboratory analytical instrument and labels or ID numbers on the sides of the containers may interfere with the analysis. Check with the laboratory for proper placement of labels or field ID numbers.
 - 3.5. Ship in an upright position in the shipping containers that have been provided by the laboratory. If none are provided, protect vials from breakage ("bubble wrap" is recommended), segregate replicate samples in separate plastic bags, and ship to the laboratory in an upright position.
4. DOCUMENTATION
 - 4.1. Complete the field notes.
 - 4.2. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2009. *Cyanide Sampling*

Cyanide is a very reactive and unstable species and is highly toxic. Samples suspected of containing cyanide must be handled very carefully.

1. SAMPLE CONTAINERS
 - 1.1. Use polyethylene or glass sample containers.
 - 1.2. Use properly cleaned containers (see FC 1300).
 - 1.3. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.
2. SAMPLE COLLECTION PROCEDURES
 - 2.1. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.
3. PRESERVATION
 - 3.1. Many different analytes interfere with the cyanide analysis (e.g., sulfides). If any interferences are known to be present, pretreat the sample for interferences by following the applicable footnotes in Table FS 1000-4.
 - 3.2. Preserve the sample within 15 minutes of sample collection.
 - 3.3. Preserve samples with sodium hydroxide to a pH greater than 12.
 - 3.4. Preserve the sample by adding an accurately measured amount of a sodium hydroxide solution or sodium hydroxide pellets to the container. Use a graduated container or pipet to add the solution.
 - 3.4.1. Tightly cap the sample container and shake to distribute the preservative.
 - 3.4.2. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is greater than 12. **Do not put the pH paper directly into the sample container.**
 - 3.4.3. If the pH is less than 12, add additional measured amounts of the preservative and test with narrow range pH paper (see section 3.4.2 above) until the pH has been raised to above 12 pH units.

- 3.4.4. Record the total amount of preservative that was added to the sample.
 - 3.5. After the sample has been preserved, screw the cap on tightly.
 - 3.6. Immediately put the sample in **wet** ice (see FS 1006, section 5).
 - 3.7. Preserve at least one of the equipment blanks with all the reagents and the **greatest** amount of sodium hydroxide that was required in the sample set and note the amount in field documentation.
4. DOCUMENTATION
- 4.1. Complete the sample container label and stick firmly on the container.
 - 4.2. Complete the field notes.
 - 4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.
 - 4.4. Ensure that all preservation measures are part of the field notes.

FS 2010 *Sulfide Sampling*

1. Analyze samples within 15 minutes of collection, or the preserve the sample within 15 minutes for later analysis. If preservation is required add the zinc acetate and sodium hydroxide to the container **before** filling with sample.
2. Avoid aerating the sample during collection. Slowly pour the sample slowly and carefully allow the sample to flow down the **side** of the container to minimize turbulence.
3. Check the pH (if necessary) before completing the filling process.
4. Complete the filling process. **Do not leave a head space.**

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 in-place 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).

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 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).

2.2.2.2 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 appropriate DEP program or project manager. DEP does not recommend using bailers because improper bailing:

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 water).

3.3.2 Record the length of the tape relative to the reference point (see section 3.2 above).

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.

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.

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 aquifer 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.

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.

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 and filters (if possible).

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 bottom of 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 the spigot, valve or other sampling point closest to the tank.
 - 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, bladder or submersible pump with the in-line filter fitted on the outlet end.

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 filtration 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

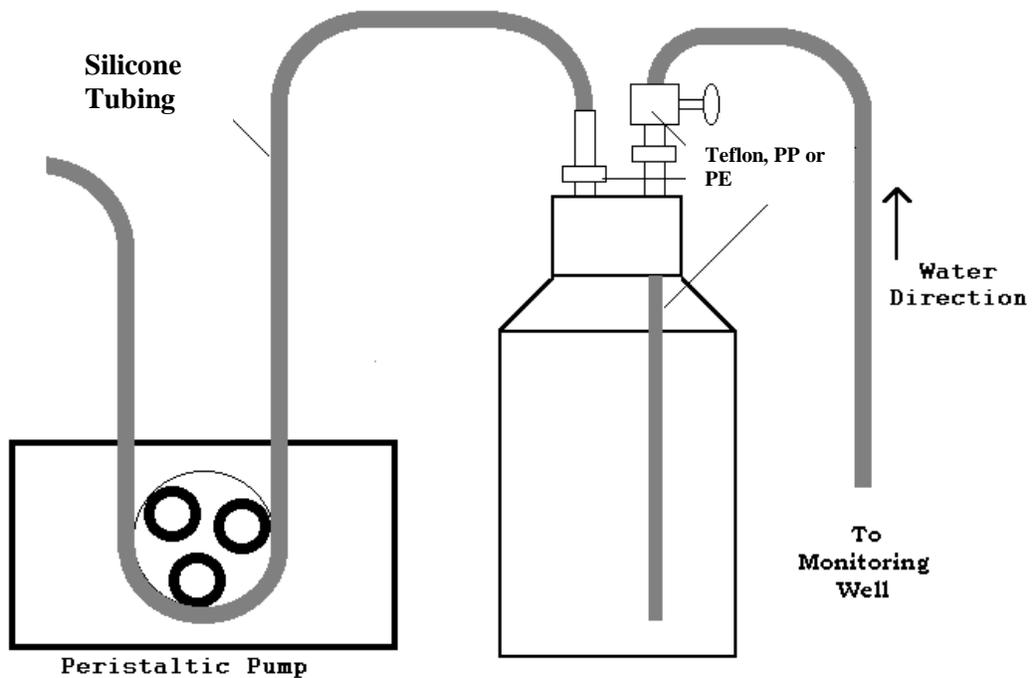
**Table FS 2200-3
 Allowable Uses for Bailers**

• 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

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)	± 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

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.

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- 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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FT 1100 Field Measurement of Hydrogen Ion Activity (pH)

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 μ mhos/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 1300. Field Measurement of Salinity

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. INTRODUCTION: Salinity is an important property of industrial and natural waters. This field parameter is also important for assessing the source or origin of effluents and of the mixing between fresh and marine waters in coastal regions, in both surface water and groundwater.

1.1. Salinity is a unit-less parameter since by definition it is the ratio of the mass of dissolved salts to the total mass of a given volume of water. Thus, salinity values are commonly expressed as “grams of salt/kilograms of water” or ‰.

1.2. Salinity is determined by using indirect methods involving the measurement of a related physical property such as conductivity, density, sound speed, or refractive index. The commonly used procedures in the field are determination of conductivity or density of the sample.

1.3. The sample salinity is calculated from an empirical relationship between salinity and the physical property as determined from a standard solution. Refer to the referenced method SM2520 for further discussions on these topics.

1.4. Because of its high sensitivity and easy of measurement, the conductivity method is most often used to determine the salinity. (Note – using a hydrometer to measure the density or the specific gravity to obtain an approximate salinity value is not recommended for reporting purposes.)

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Depending on the chosen method, use:

2.1.1. Any self-contained conductivity instrument with a platinum or graphite electrode type cell, and a temperature sensor. Some conductivity instruments have meter scales pre-calibrated for salinity and are sometimes referred to as Salinometers. For routine fieldwork use a conductivity meter accurate and reproducible to at least 5% or 1 $\mu\text{mho/cm}$ (whichever is greater), and equipped with temperature-compensation adjustment; or

2.1.2. A precision “vibrating flow densimeter” (see Millero & Poisson, 1981) and a field thermometer.

2.2. Standards:

2.2.1. Purchased or laboratory-prepared Standard Seawater and/or potassium chloride (KCl) standards of appropriate equivalent salinities.

2.2.1.1. In the laboratory, prepare the Standard Seawater per recipe in method SM2520 and SM8010 (Table III), and standard KCl solutions per recipe in method SM2510 (American Public Health Association, American Water Works Association, Water Pollution Control Federation, Standard Methods for the Examination of Water and Wastewater).

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2.2.2. De-ionized water for calibration of the densimeter (if used).

2.3. Recordkeeping and Documentation Supplies:

- Field logbook (w/ waterproof paper is recommended) or field forms
- Indelible pens

3. CALIBRATION AND USE

3.1. Conductivity Method

3.1.1. Calibration: - Calibrate the instrument per manufacturer's instructions using one calibration standard, either standard seawater or a KCl solution, as applicable. The acceptance criterion for initial calibration or a calibration verification is that the instrument reading is within +/- 5% of the standard value. For example, when calibrating with standard seawater, $S = 35$, the meter must read in the 34 to 36 range in order to be acceptable.

3.1.1.1. Use standard seawater ($S = 35$) when measuring salinity in the open ocean or estuaries with a predominance of seawater.

3.1.1.2. KCl may be used in estuarine waters with low salinity ($S = 0 - 40$).

3.1.1.3. If verifying or calibrating with a "zero" standard, do not use analyte-free water or air check (dry electrode) as the blank.

3.1.1.4. If the meter does not provide a direct reading of salinity, use the equation found in SM2520B to convert the readings to salinity.

3.1.1.5. Follow the calibration activities in FT 1000, section 2.2.

3.1.1.6. Do not reuse standards for initial calibrations.

3.1.2. Field Use: - Rinse the probe with DI water after calibration and before each sample measurements. Follow the manufacturer's instructions for temperature compensation, if needed. Report salinities with only one decimal figure.

3.1.3. General Concerns for Conductivity Method

3.1.3.1. 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.

3.1.3.2. Thoroughly rinse the conductivity (salinity) 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.

3.1.3.3. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.3.4. 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

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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. Density Method

The vibrating flow densimeter is an instrument that allows for precise and rapid measurements of the density of a liquid, such as water. The principle of operation is the effect of the density of the sample on the frequency of a vibrating tube encased in a constant-temperature jacket. The measurement is made by passing the water (sample) through the vibrating tube and reading the period of vibration that is electronically sensed and displayed by the densimeter. The sample density (D) is proportional to the square of the period of vibration (T):

$$D = a + bT^2$$

Where a and b are terms determined by calibration, b being determined by calibration of the densimeter with Standard Seawater. The difference between the density of the sample (D) and that of pure water (D₀) is given by:

$$D - D_0 = b (T^2 - T_0^2)$$

Where T and T₀ are, respectively, the periods of the sample and that of pure (de-ionized) water. Using this second equation, you only have to deal with the term b for calibration purposes. Hence, the system can be calibrated with two liquids: pure water and Standard Seawater. Follow the manufacturer's instruction for calibration of the densimeter.

The salinity of the sample is determined by the one-atmosphere international equation of state for seawater. This equation relates the difference (D - D₀) to the practical salinity as a function of the temperature of the sample (which is also measured by the densimeter or the field thermometer). For further details on this calculation read the referenced method SM2520C.

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.

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- 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.
 - 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., salinity standard)
 - Value of standard, including correct units (e.g., salinity = 20 ‰)
 - 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)

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- 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.

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 calendar frequency.**

2.5.1.2. For an accuracy calibration verification 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 or instrument 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.

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- 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.

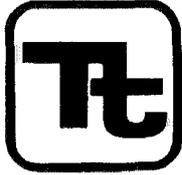
3.5. Measuring Turbidity in Samples

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.

DEP-SOP-001/01
FT 1600 Field Measurement of Turbidity

- 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 06/99	Revision 1
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject
BOREHOLE AND SAMPLE LOGGING

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1 (CONTINUED)

SOIL TERMS

FINE-GRAINED SOILS More Than Half of Material is Finer Than No. 200 Sieve Size		FINE-GRAINED SOILS More Than Half of Material is Finer Than No. 200 Sieve Size	
GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION
OH	Very high plasticity	OH	Very high plasticity
SH	High plasticity	SH	High plasticity
ML	Medium plasticity	ML	Medium plasticity
CL	Low plasticity	CL	Low plasticity
OL	Very low plasticity	OL	Very low plasticity

DENSITY OF GRANULAR SOILS

RELATION	SYMBOL	DESCRIPTION
Very Loose	VL	
Loose	L	
Medium Dense	MD	
Dense	D	
Very Dense	VD	

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNSATURATED WATER CONTENT (%)	LIQUID LIMIT (%)	PLASTICITY INDEX	FIELD IDENTIFICATION NOTES
Very Stiff	Less than 15	10-12	1-2	Usually contained below 10cm by 10
Stiff	15-20	12-14	2-4	Usually contained in some holes by 10cm
Medium Stiff	20-25	14-16	4-6	Can be penetrated from 10cm by 10cm
Soft	25-30	16-18	6-8	Usually penetrated by 10cm
Very Soft	30-40	18-20	8-10	Usually penetrated by 10cm
Flowing	Over 40	Over 20	Over 10	Flowing in all directions

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)

ROCK TYPE	DESCRIPTION	ROCK TYPE	DESCRIPTION
Very Hard	Harder than 1000 kg/cm ²	Very Hard	Harder than 1000 kg/cm ²
Hard	Harder than 500 kg/cm ²	Hard	Harder than 500 kg/cm ²
Medium Hard	Harder than 250 kg/cm ²	Medium Hard	Harder than 250 kg/cm ²
Soft	Harder than 100 kg/cm ²	Soft	Harder than 100 kg/cm ²
Very Soft	Harder than 50 kg/cm ²	Very Soft	Harder than 50 kg/cm ²

LEGEND:

SOIL SAMPLES - TESTS

14" Standard Sample
 8" x 8" Standard Sample
 0 - Other Sample, Specify in Remarks

ROCK SAMPLES - TESTS

XMC (Compressive Core), 14" x 14"
 OMC (Ombro Core), 14" x 14"
 2 - Other Core Tests, Specify in Remarks

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5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inch Φ -1/2 inch Φ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

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Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

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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

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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 (CaCO₃). 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'± SET TEMP 6" CAS TO 15.5				
	20.0			20.0									
	21.0			21.0									
	22.0			22.0									
	23.0			23.0									
	24.0			24.0									
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	98.0			98.0									
	99.0			99.0									
	100.0			100.0									

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

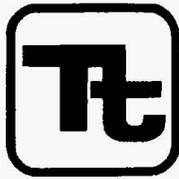
Unified Soil Classification System (USCS).

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 id 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.

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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

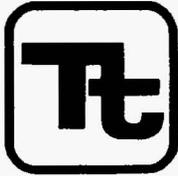
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ATTACHMENT B

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**

<p>Alabama Alabama One-Call 1-800-292-8525</p> <p>Alaska Locate Call Center of Alaska, Inc. 1-800-478-3121</p> <p>Arizona Arizona Blue Stake 1-800-782-5348</p> <p>Arkansas Arkansas One Call System, Inc. 1-800-482-8998</p> <p>California Underground Service Alert North 1-800-227-2600 Underground Service Alert of Southern California 1-800-227-2600</p> <p>Colorado Utility Notification Center of Colorado 1-800-922-1987</p> <p>Connecticut Call Before You Dig 1-800-922-4455</p> <p>Delaware Miss Utility of Delmarva 1-800-282-8555</p> <p>Florida Sunshine State One-Call of Florida, Inc. 1-800-432-4770</p> <p>Georgia Underground Protection Center, Inc. 1-800-282-7411</p> <p>Hawaii Underground Service Alert North 1-800-227-2600</p> <p>Idaho Dig Line Inc. 1-800-342-1585 Kootenai County One-Call 1-800-428-4950 Shoshone - Benewah One-Call 1-800-398-3285</p> <p>Illinois JULIE, Inc. 1-800-892-0123 Digger (Chicago Utility Alert Network) 312-744-7000</p> <p>Indiana Indiana Underground Plant Protection Service 1-800-382-5544</p>	<p>Iowa Iowa One-Call 1-800-292-8989</p> <p>Kansas Kansas One-Call System, Inc. 1-800-344-7233</p> <p>Kentucky Kentucky Underground Protection Inc. 1-800-752-6007</p> <p>Louisiana Louisiana One Call System, Inc. 1-800-272-3020</p> <p>Maine Dig Safe System, Inc. 1-888-344-7233</p> <p>Maryland Miss Utility 1-800-257-7777 Miss Utility of Delmarva 1-800-282-8555</p> <p>Massachusetts Dig Safe System, Inc. 1-888-344-7233</p> <p>Michigan Miss Dig System, Inc. 1-800-482-7171</p> <p>Minnesota Gopher State One Call 1-800-252-1168</p> <p>Mississippi Mississippi One-Call System, Inc. 1-800-227-6477</p> <p>Missouri Missouri One-Call System, Inc. 1-800-344-7483</p> <p>Montana Utilities Underground Protection Center 1-800-424-5555 Montana One Call Center 1-800-551-8344</p> <p>Nebraska Diggers Hotline of Nebraska 1-800-331-5666</p> <p>Nevada Underground Service Alert North 1-800-227-2600</p> <p>New Hampshire Dig Safe System, Inc. 1-888-344-7233</p>	<p>New Jersey New Jersey One Call 1-800-272-1000</p> <p>New Mexico New Mexico One Call System, Inc. 1-800-321-2537 Las Cruces- Dona Ana Blue Stakes 1-888-526-0400</p> <p>New York Dig Safely New York 1-800-862-7962 New York City- Long Island One Call Center 1-800-272-4480</p> <p>North Carolina The North Carolina One-Call Center, Inc. 1-800-632-4949</p> <p>North Dakota North Dakota One-Call 1-800-795-0555</p> <p>Ohio Ohio Utilities Protection Service 1-800-362-2764 Oil & Gas Producers Underground Protect'n Svc 1-800-925-0988</p> <p>Oklahoma Call Okie 1-800-522-6543</p> <p>Oregon Oregon Utility Notification Center/One Call Concepts 1-800-332-2344</p> <p>Pennsylvania Pennsylvania One Call System, Inc. 1-800-242-1776</p> <p>Rhode Island Dig Safe System, Inc. 1-888-344-7233</p> <p>South Carolina Palmetto Utility Protection Service Inc. 1-888-721-7877</p> <p>South Dakota South Dakota One Call 1-800-781-7474</p> <p>Tennessee Tennessee One-Call System, Inc. 1-800-351-1111</p>
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ATTACHMENT 1 (Continued)

Texas

Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah

Blue Stakes of Utah
1-800-662-4111

Vermont

Dig Safe System, Inc.
1-888-344-7233

Virginia

Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington

Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating
Council
509-456-8000

West Virginia

Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin

Diggers Hotline, Inc.
1-800-242-8511

Wyoming

Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia

Miss Utility
1-800-257-7777

Alberta

Alberta One-Call Corporation
1-800-242-3447

British Columbia

BC One Call
1-800-474-6886

Ontario

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

Quebec

Info-Excavation
1-800-663-9228

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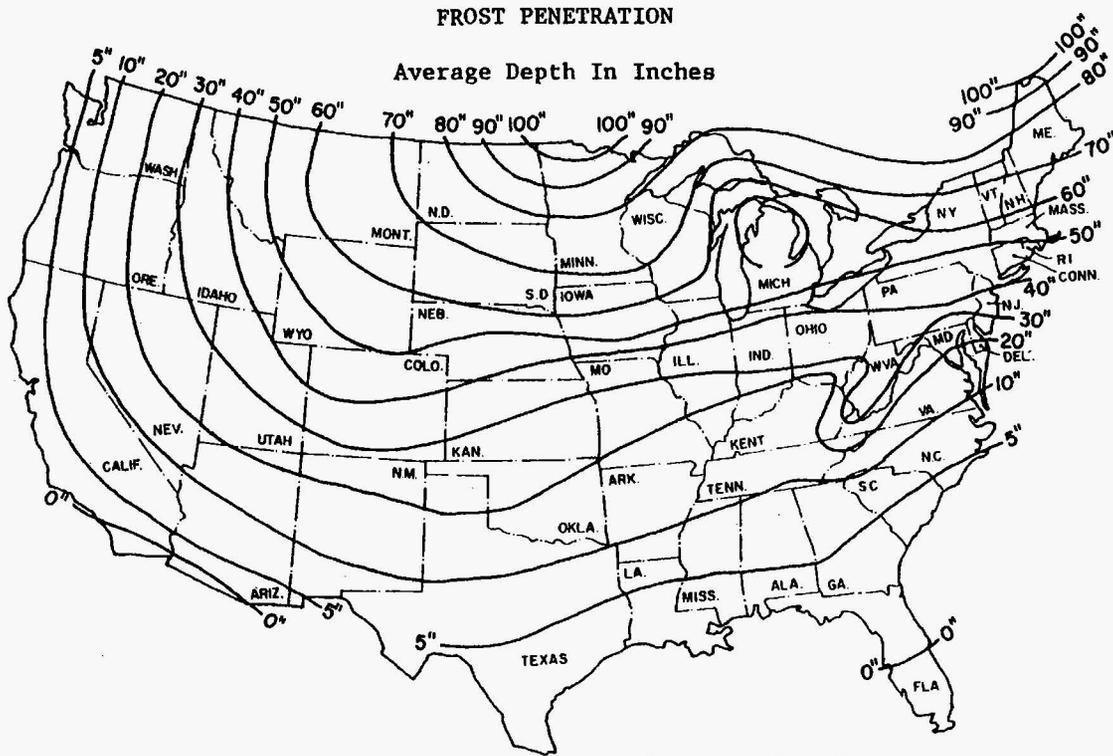
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ATTACHMENT 2

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

STANDARD OPERATING PROCEDURES

Number	SA-1.1	Page	1 of 34
Effective Date	04/07/2008	Revision	7
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject
GROUNDWATER SAMPLE ACQUISITION AND
ONSITE WATER QUALITY TESTING

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

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Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol (⁰/₀₀) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

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- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

Knee injuries – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.

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- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- **Face Traffic.** Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

CAUTION

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

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Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

CAUTION

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.
2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.
3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

CAUTION

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:
 - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.
 - While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

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- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
 - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
 - pH Paper
 - Camera and film (if appropriate)
 - Appropriate keys (for locked wells)
 - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
 - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
 - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.
- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

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6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

where: V = Static volume of well in gallons.
T = Linear feet of water in the well.
r = Inside radius of well casing in inches.
0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

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6.4 Evacuation of Static Water – Purging

6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

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Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.

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- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

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To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters

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used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

6.5.1 Measurement of pH

6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)

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- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

6.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

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2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

6.5.2 Measurement of Specific Conductance

6.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

6.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

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6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

6.5.3 Measurement of Temperature

6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

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6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.
2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.
2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.
3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

6.5.4 Measurement of Dissolved Oxygen

6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

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the two metals, reduction of oxygen to hydroxide ion (OH⁻) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

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7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.
8. Rinse the probe with deionized water.
9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

6.5.5 Measurement of Oxidation-Reduction Potential

6.5.5.1 General

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

6.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

6.5.5.3 Equipment

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

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2. Thoroughly rinse the electrode with deionized water.
3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

6.5.6 Measurement of Salinity

6.5.6.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

6.5.6.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

6.5.6.3 Equipment

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.6.4 Measurement Techniques for Salinity

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the meter before going into the field.

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3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.
5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the probes with deionized water.

6.5.7 Measurement of Turbidity

6.5.7.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

6.5.7.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

6.5.7.3 Equipment

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

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6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

6.6 Sampling

6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

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Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:
 - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
 - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
 - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

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during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.
4. Calculate volume of well water to be removed as described in Section 6.3.
5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.
6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.
7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.
8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.
10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.
11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.
12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

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occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.
14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.
15. Process sample containers as described in SOP SA-6.1.
16. Decontaminate equipment as described in SOP SA-7.1.

6.7 Low-Flow Purging and Sampling

6.7.1 Scope and Application

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

6.7.2 Equipment

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

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- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).
2. Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.
3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.
4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well
5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

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6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.
7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.
9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.
10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.
11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:
 - pH ± 0.2 standard units
 - Specific conductance $\pm 10\%$
 - Temperature $\pm 10\%$
 - Turbidity less than 10 NTUs
 - DO $\pm 10\%$
12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.

NOTE: VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

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- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.
- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.
- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.
- Prepare samples for shipping as per SOP SA-6.1.

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ATTACHMENT A
PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A
PURGING EQUIPMENT SELECTION
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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/L length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	\$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	\$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	\$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	\$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	\$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	\$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	\$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	\$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	\$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	\$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	\$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	\$300-400	Requires compressed gas; piezometric level indicator; other materials available.

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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/L ength (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	\$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	\$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	\$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	\$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	\$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	\$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	\$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

PE Polyethylene
 PP Polypropylene
 PVC Polyvinyl chloride
 SS Stainless steel
 PC Polycarbonate
 EPDM Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

NA Not applicable
 AC Alternating current
 DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

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STANDARD OPERATING PROCEDURES

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Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject
SOIL SAMPLING

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

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Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

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- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

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- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket.**

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6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 **Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis**

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

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obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.
2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.
3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.
4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.
5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.
6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.
7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.
8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.
9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.
10. Label the bag with appropriate information in accordance with SOP SA-6.3.
11. Place the full sampler inside a lined cooler with ice and cool to 4°C ± 2 °C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.
12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.
13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™ samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

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6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.
2. Pull the plunger back and insert the syringe into the soil to be sampled.
3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.
4. Weigh the sample and adjust until obtaining the required amount of sample.

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5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.
6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.
7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.
8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.
9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

CAUTION

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.
2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation
3. Add the weighed sample to the sample vial.
4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.
5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

NOTE

If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

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1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.
2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.
3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.
4. Transfer the mixed soil to the appropriate sample containers and close the containers.
5. Label the sample containers in accordance with SOP SA-6.3.
6. Place the containers in a cooler of ice as soon after collection as possible.
7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

NOTE

Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4° C.

NOTE

Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

NOTE

Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:
 - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

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REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
 - Review the Safe Work Permit prior to conducting the activity.
 - Review the activity to be conducted.
2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.
4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.
5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.
6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.
7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.
8. Remove disturbed material in the upper end of the tube and measure the length of sample again.
9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

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10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.
11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.
12. Mark the "up" direction on the side and upper end of the tube with indelible ink.
13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).
14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.
- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

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6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

NOTE

Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.

For the purposes of instruction, the terms “surface soil” and “near-surface soil” are used in this SOP as follows:

- Surface soil - 0 to 6 inches bgs
- Near-surface soil - 6 to 18 inches bgs

If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
 - Nitrile surgeon’s or latex gloves may be used, layered as necessary.
 - Safety glasses
 - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

Safety Reminder

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

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- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.
2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

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3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be preceded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

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5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.
6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.
7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.
8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.
9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.
10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.
11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.
13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.
14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such as the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

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- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

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2. Lower the sampler into the borehole inside the hollow stem auger bits.
3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.
4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.
5. Detach the sampler from the drill rods.
6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

CAUTION

Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.
8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.
9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings were encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).
10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.
11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).
12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

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6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 **Applicability**

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 **Test Pit and Trench Excavation**

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration

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- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example,

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samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

6.8.3.2 Sampling Equipment

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

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- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
 - the operator shall wait while the sampler inspects the test pit from grade level
 - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
 - Any fluid phase, including groundwater seepage, is encountered in the test pit
 - Any drums, other potential waste containers, obstructions, or utility lines are encountered
 - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

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- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:
 - a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.
 - b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.
 - c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.
 - d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.
 - e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.
 - f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.
- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

CAUTION

Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:
 - a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.
 - b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.
 - c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.
- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

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6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable through soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e.g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

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Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

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If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O₂ meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

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- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

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

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



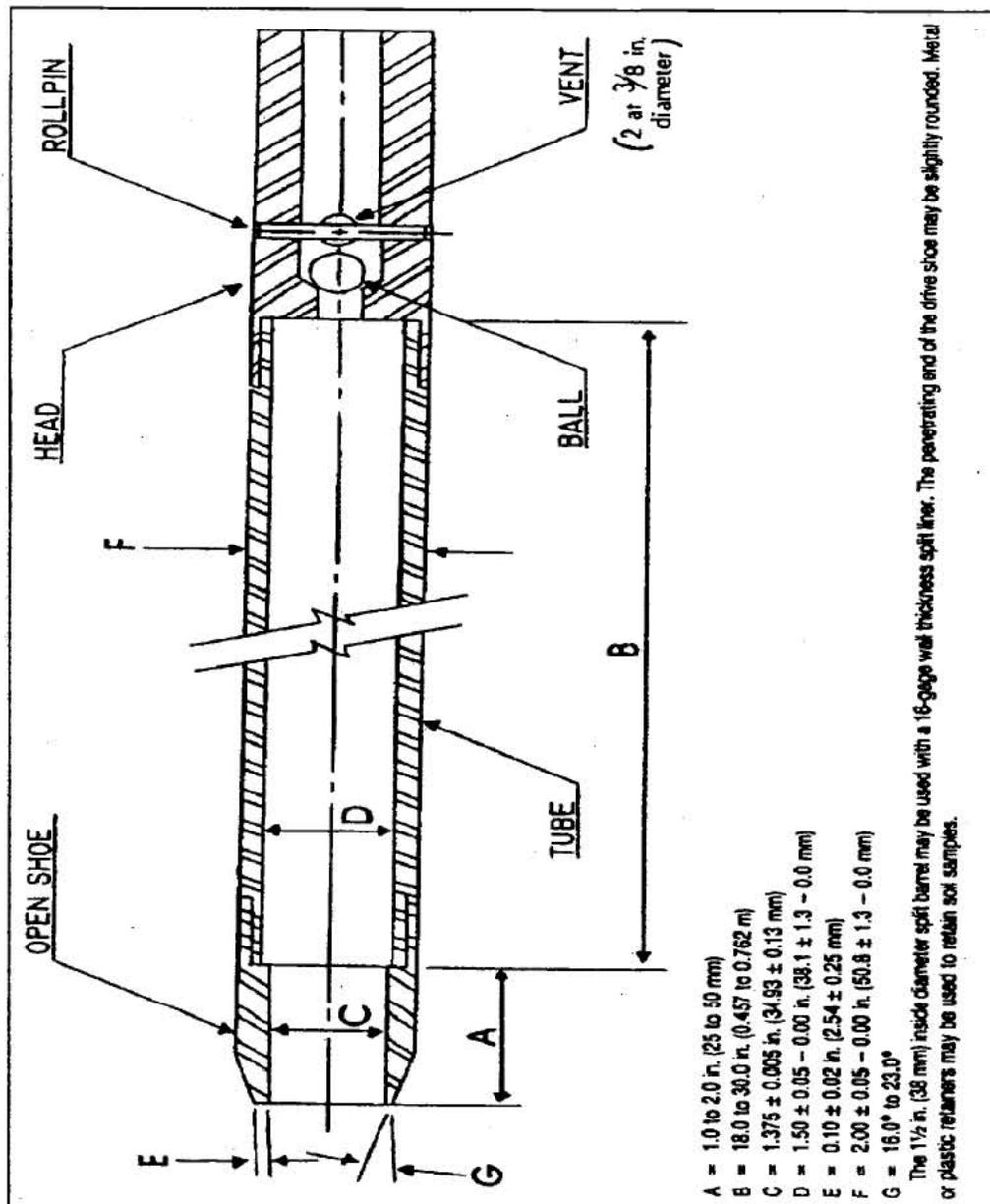
Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

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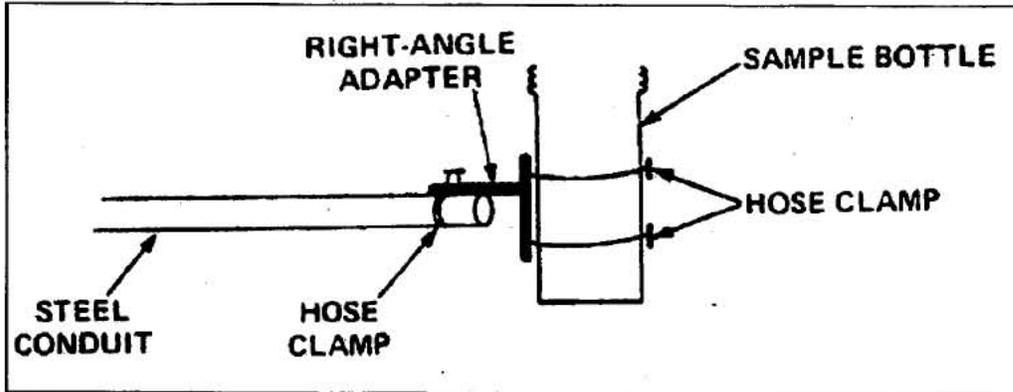
Project Site Name: _____		Sample ID No.: _____		
Project No.: _____		Sample Location: _____		
<input type="checkbox"/> Surface Soil		Sampled By: _____		
<input type="checkbox"/> Subsurface Soil		C.O.C. No.: _____		
<input type="checkbox"/> Sediment		Type of Sample:		
<input type="checkbox"/> Other: _____		<input type="checkbox"/> Low Concentration		
<input type="checkbox"/> QA Sample Type: _____		<input type="checkbox"/> High Concentration		
GRAB SAMPLE DATA:				
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)	
Time:				
Method:				
Monitor Reading (ppm):				
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				
SAMPLE COLLECTION INFORMATION:				
Analysis	Container Requirements	Collected	Other	
OBSERVATIONS / NOTES:		MAP:		
Circle if Applicable:		Signature(s):		
MS/MSD	Duplicate ID No.:			

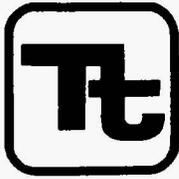
ATTACHMENT B SPLIT-SPOON SAMPLER



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





TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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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	Revision 3	Effective Date 09/03

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 Yes No
- Physical hazards removed or blockaded Yes No
- Site control boundaries demarcated/signage Yes No

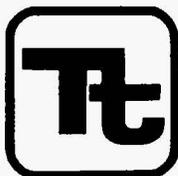
X. Equipment Preparation

- | | | |
|------------------------------------------------------------|------------------------------|----------------------------------------|
| Equipment drained/depressurized..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Equipment purged/cleaned..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Isolation checklist completed..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Electrical lockout required/field switch tested..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Blinds/misalignments/blocks & bleeds in place..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Hazardous materials on walls/behind liners considered..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |

- XI. Additional Permits required (Hot work, confined space entry). Yes No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	SA-6.1	Page	1 of 11
Effective Date	02/04	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject
NON-RADIOLOGICAL SAMPLE HANDLING

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

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

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

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

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

n.o.s. - Not otherwise specified.

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

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

Common Preservatives:

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

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Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na₂S₂O₃

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

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

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

4.0 RESPONSIBILITIES

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

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

5.0 PROCEDURES

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

5.1 Sample Containers

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

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

5.2 Sample Preservation

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

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

5.2.1 Overview

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

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

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

5.2.2 Preparation and Addition of Reagents

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

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

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

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

Additional considerations are discussed below:

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

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

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

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

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

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

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

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

Continue with proper acidification of the sample as described above.

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

5.3 Field Filtration

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

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

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

5.4 Sample Packaging and Shipping

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

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

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

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

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

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

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

6.0 REFERENCES

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

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

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

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

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

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ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

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

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

SOIL

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

AIR

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

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

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ATTACHMENT B

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

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

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

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

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

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

METALS:⁽⁷⁾

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

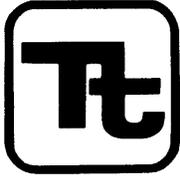
ORGANIC TESTS:⁽⁸⁾

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

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

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



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

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Effective Date	03/09/09	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject
FIELD DOCUMENTATION

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

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

General personnel qualifications for field documentation activities include the following:

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

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

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- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

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separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (<http://intranet.ttnus.com>) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc[®]-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

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and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 **Hydrogeological and Geotechnical Forms**

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

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5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 **Equipment Calibration and Maintenance Form**

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 **FIELD REPORTS**

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

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5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

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

In addition to those described herein, other summary reports may also be contractually required.

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

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

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- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

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**ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY**

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL: _____

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ____ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

Field Operations Leader

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**ATTACHMENT B
SAMPLE LABEL**

	Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
			Site:
		Location:	
Sample No:		Matrix:	
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	



TETRA TECH NUS, INC.

CHAIN OF CUSTODY

NUMBER 3413

PAGE ___ OF ___

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

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY) 4/02R FORM NO. TINUS-001

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ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM

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**ATTACHMENT D
CHAIN-OF-CUSTODY SEAL**

<u>Signature</u> <hr/> <u>Date</u> <hr/> CUSTODY SEAL		CUSTODY SEAL <hr/> <u>Date</u> <hr/> <u>Signature</u>
--------------------------------------------------------------------	--	--------------------------------------------------------------------



STANDARD OPERATING PROCEDURES

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Effective Date 01/28/2009	Revision 6
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment, chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent - A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

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4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

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

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

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- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

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- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:
 - Well removed from pedestrian/vehicle thoroughfares.
 - Avoidance of areas where control/custody cannot be maintained.
 - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
 - Avoidance of potentially contaminated areas.
 - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

The selected decontamination site should include the following, where possible:

- Areas where potable water and electricity are provided.

Safety Reminder

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).
- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

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- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.
- Areas that offer sufficient size to carry out the specific decontamination sequence.
- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:
 - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.
 - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.
 - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.
 - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.
 - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:
 - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

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- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.
- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.
2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.
3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.
4. Remove the pump and tubing/bailer from the container
5. Rinse external pump components using tap water.

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6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents –
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.
8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.
9. Drain residual deionized water to the extent possible.
10. Allow components of the equipment to air dry.
11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.
12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

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7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.
- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:
 1. Wash with soap and water
 2. Rinse with tap water
 3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 **Downhole Drilling Equipment**

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

CAUTION

Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

CAUTION

In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

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4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol
5. To the extent possible, allow components to air dry.
6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.
7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.
2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.
3. Always wear PPE as specified in the HASP such as:
 - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.
4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.
5. Do not modify equipment unless the manufacturer has approved the modifications.

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7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

CAUTION

When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

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7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

NOTE

Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

NOTE

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).
4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:
 - Enclose areas accessible by the general public using construction fencing and signs.
 - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
 - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
 - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
 - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
 - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

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- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

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CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.
- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:
 - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
 - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.
 - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:
 - Per decontamination method
 - Per disposable article/batch number of disposable articles

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NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.



STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject DECONTAMINATION OF FIELD EQUIPMENT

Attachment A iDW Label

INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE _____ JOB NO. _____

LOCATION _____

DATE _____

DRUM# _____

CONTENTS _____

VOLUME _____

CONTACT _____

EMERGENCY PHONE NUMBER _____

STANDARD OPERATING PROCEDURE

SOP-01

GLOBAL POSITIONING SYSTEM

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and update existing Geographic Information System (GIS) data. This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXM or GeoXH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at <http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf>

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific shape files, data points, background images, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS, Inc.
Attn: John Wright
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 Required GPS Hardware

- Hand-held GPS Unit capable of sub-meter accuracy (i.e. Trimble GeoXM or Trimble GeoXH). This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer.

Optional Accessories:

- External antenna
 - Range pole
 - Hardware clamp (for mounting Geo to range pole)
 - GeoBeacon
- Indelible marker
 - Non-metallic pin flags for temporary marking of positions

2.2 Required GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)
- Microsoft ActiveSync version 4.2 or later. Download to personal computer from:
http://www.microsoft.com/windowsmobile/en-us/downloads/eulas/eula_activesync45_1033.mspx?ProductID=76
- Trimble Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from:
<http://www.trimble.com/datatransfer.shtml>

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

Start Up:

- 1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.
- 2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.
- 3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.
- 4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.1 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup.

- 1) Coordinate System
 - a. Tap on the Coordinate System.
 - b. Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
Note: It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
 - c. Tap on the Units.
 - d. Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - e. Tap Real-time Settings.
 - f. Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - g. The GPS unit is now configured correctly for your specific project.

4.0 ANTENNA CONNECTION

- 1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
- 2) At this point the GPS unit is ready to begin collecting data.

5.0 COLLECTING NEW DATA IN THE FIELD

- 1) From the Main Menu select Data.
- 2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
- 3) An auto-generated filename appears and should be edited for your specific project. If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.
- 4) After entering the file name, tap Create to create the new file.
- 5) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet).
- 6) The Choose Feature screen appears.

5.1 Collecting Features

- 1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
- 2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**
- 3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
- 4) Upon arriving at the specific location, tap on Point_generic as the Feature Name.
- 5) Tap Create to begin data logging.
- 6) In the Comment Box enter sample ID or location-specific information.
- 7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
- 8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.
- 9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2.

5.2 Viewing Data or Entering Additional Data Points to the Current File

- 1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
- 2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

5.3 Viewing Data or Entering Data Points from an Existing File

- 1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
- 4) Highlight the file you want to view and select Map from the Main Menu.
- 5) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

6.0 NAVIGATION

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

- 1) From the Main Menu select Map.
- 2) Using the Select tool, pick the point on the map to where you want to navigate.
- 3) The location you select will have a box placed around the point.
- 4) From the Options menu, choose the Set Nav Target (aka set navigation target).
- 5) The location will now have double blue flags indicating this point is you navigation target.
- 6) From the Main Menu select Navigation.

- 7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.
- 8) Follow the navigation guide until you reach the point you select.
- 9) Repeat as needed for any map point by going back to Step 1.

7.0 PULLING IN A BACKGROUND FILE

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

- 1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.
- 2) Select the project-specific background file from the list of available files.
- 3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/> functions at the bottom of the screen.
- 4) In operating mode, the operator's location will show up on the background file as a floating "X".

8.0 DATA TRANSFER

This section provides instructions on how to transfer stored data on the handheld GPS unit to a personal computer. Prior to transferring data from the GPS unit to a computer, Microsoft ActiveSync and Trimble Data Transfer Utility software must be downloaded to the computer from the links provided in Section 2.2 (Required GPS Software). If a leased computer is utilized in which the operator can not download files, see the Note at the end of Section 8.0.

- 1) See Attachment A at the end of this SOP for instructions on how to transfer data from the GPS to a personal computer.

Note: If you are unable to properly transfer data from the GPS unit to a personal computer, the unit should be shipped to the project-specific contact listed in Section 1.0 where the data will be transferred and the GPS unit then shipped back to the vendor.

9.0 SHUTTING DOWN

This section provides instruction for properly shutting down the GPS unit.

- 1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.

- 2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
- 3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.
- 4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

ATTACHMENT A

How to Transfer Trimble GPS Data between Data Collector and PC

original 11/21/06 (5/1/08 update) – John Wright

Remember – Coordinate System, Datum, and Units are critical!!!

Trimble Data Collection Devices:

Standard rental systems include the Trimble ProXR/XRS backpack and the newer handheld GeoXT or GeoXH units. Some of the older backpack system may come with either a RECON “PDA-style” or a TSCe or TSC1 alpha-numeric style data collector.

The software on all of the above units should be Trimble TerraSync (v 2.53 or higher – current version is 3.20) and to the user should basically look and function similar. The newer units and software versions (which should always be requested when renting) include enhancements for data processing, real-time display functions, and other features.

Data Transfer:

Trimble provides a free transfer utility program to aid in the transfer of GIS and field data. The Data Transfer Utility is a standalone program that will run on a standard office PC or laptop.

To connect a field data collector such as a RECON, GeoXM, GeoXT, GeoXH, or ProXH, you must first have Microsoft ActiveSync installed to allow the PC and the data collector to talk to one another. A standard USB cable is also needed to connect the two devices.

A CD or USB drive is provided with the data collector for use in data transfer. If needed, these programs are also available without charge via the web at:

- **Trimble Data Transfer Utility** (v 1.38) program to download the RECON or GeoXH field data to your PC: <http://www.trimble.com/datatransfer.shtml>
- **ActiveSync** from Microsoft to connect the data collector to the PC. The latest version (v4.5) can be found at: <http://www.microsoft.com/windowsmobile/activesync/default.mspx>
(see page 2 for data transfer instructions)

To Transfer Data Collected in the Field:

- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Make sure the data file desired is CLOSED in TerraSync prior to transfer
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Receive**" data tab (under device)
- Select "**Data**" from file types on the right
- Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
- Select or browse to a C-drive folder you can put this file for emailing
- When the file appears on the list, hit the "**Transfer All**"
- Go to your Outlook or other email, send a message to: John.Wright@tetrattech.com (or GIS department)
- Attach the file(s) you downloaded from your C-drive. For each TerraSync data file created you should have a packet of multiple data files. All need to be sent as a group – make sure you attach all files (the number of files may vary – examples include: ssf, obx, obs, gix, giw, gis, gip, gic, dd, and car)

To Transfer GIS Data from PC to the Field Device (must be converted in Pathfinder Office):

- Obtain GIS file(s) desired from GIS Department and have converted to Trimble extension
- Contact John Wright (John.Wright@tetrattech.com) if needed for file conversion and upload support
- The GIS file(s) can be quickly converted if requested and sent back to the field user in the needed "Trimble xxx.imp" extension via email – then quickly downloaded from Outlook to your PC for transfer
- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Send**" data tab (under device)
- Select "**Data**" from file types on the right (you can also send background files)
- Browse to the location of the data on your PC (obtain the file from Pathfinder Office or from the person who converted the data for field use)
- Select the options as appropriate for the name and location of the data file to go on the data collector (usually you can choose main memory or a data storage card)
- When the file(s) appears on the list, hit the "**Transfer All**"
- Run TerraSync on the field device and open the existing data files. Your transferred file should appear (make sure you have selected Main Memory, Default, or Storage Card as appropriate)

APPENDIX B

**LABORATORY STANDARD OPERATING PROCEDURES
AND ACCREDITATIONS**

Empirical SOPs and Accreditations



**LABORATORY
ACCREDITATION
BUREAU**

Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2226

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228

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

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

Accreditation Granted through: November 30, 2012

**R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 30th of November 2009**

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

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

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	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	t-Butyl alcohol
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene

Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese

Non-Potable Water		
Technology	Method	Analyte
CVAA	EPA 6010B/C	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
Titration	SM 2320B 20 th /21 st edition	Alkalinity
Colorimetric	SM 4500 B, G, 20 th /21 st edition	Ammonia
UV/Vis	EPA 7196A	Hexavalent Chromium
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20 th /21 st edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20 th /21 st edition	Total Phosphorus (as P)

Non-Potable Water		
Technology	Method	Analyte
UV/Vis	SM 4500 PE, 20 th /21 st edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20 th /21 st edition	Total Organic Carbon
Gravimetric	SM 2540C, 20 th /21 st edition	TDS
Colorimetric	EPA 9012A/B	Cyanide
Physical	EPA 1010A	Ignitability
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330A	PETN
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
HPLC/MS	EPA 6850	Perchlorate

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 9012A/B	Cyanide

Solid and Chemical Materials		
Technology	Method	Analyte
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540B 20 th /21 st edition	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: _____



R. Douglas Leonard
Chief Technical Officer

Date: January 25, 2011

Issued: 11/30/09

Revised: 2/9/10

Revised: 3/31/10

Revised: 10/8/10

Revised: 1/25/11



Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

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	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	t-Butyl alcohol
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPPP (Mecoprop)
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene



Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese



Non-Potable Water		
Technology	Method	Analyte
CVAA	EPA 6010B/C	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
Titration	SM 2320B 20 th /21 st edition	Alkalinity
Colorimetric	SM 4500 B, G, 20 th /21 st edition	Ammonia
UV/Vis	EPA 7196A	Hexavalent Chromium
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20 th /21 st edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20 th /21 st edition	Total Phosphorus (as P)



Non-Potable Water		
Technology	Method	Analyte
UV/Vis	SM 4500 PE, 20 th /21 st edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20 th /21 st edition	Total Organic Carbon
Gravimetric	SM 2540C, 20 th /21 st edition	TDS
Colorimetric	EPA 9012A/B	Cyanide
Physical	EPA 1010A	Ignitability
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)



Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330A	PETN
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
HPLC/MS	EPA 6850	Perchlorate



Solid and Chemical Materials		
Technology	Method	Analyte
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 9012A/B	Cyanide



Solid and Chemical Materials		
Technology	Method	Analyte
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540B 20 th /21 st edition	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: _____

R. Douglas Leonard
Chief Technical Officer

Date: January 25, 2011

Issued: 11/30/09

Revised: 2/9/10

Revised: 3/31/10

Revised: 10/8/10

Revised: 1/25/11



State of Florida
 Department of Health, Bureau of Laboratories
 This is to certify that
 E87646

EMPIRICAL LABORATORIES, LLC.
 621 MAINSTREAM DRIVE SUITE 270
 NASHVILLE, TN 37228

has complied with Florida Administrative Code 64E-1,
 for the examination of Environmental samples in the following categories

NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS,
 NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - TOXICITY, NON-POTABLE WATER - VOLATILE ORGANICS,
 SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND
 CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, 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.

EFFECTIVE July 01, 2010 THROUGH June 30, 2011



Max Salfinger, M.D.
 Chief, Bureau of Laboratories
 Florida Department of Health
 DH Form 1697, 7/04

NON-TRANSFERABLE E87646-12-07/01/2010
 Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87646-12, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87646 EPA Lab Code: TN00004 (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	8/15/2007
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	8/15/2007
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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State Laboratory ID: E87646 EPA Lab Code: TN00004 (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	4/6/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	10/5/2006
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	4/6/2004
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Hexanone	EPA 8260	Volatile Organics	NELAP	4/6/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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State Laboratory ID: **E87646** EPA Lab Code: **TN00004** (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	10/5/2006
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/6/2004
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/6/2004
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/5/2006
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	10/5/2006
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004

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E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	10/5/2006
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acetone	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	4/6/2004
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	3/4/2008
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aluminum	EPA 200.7	Metals	NELAP	4/6/2004
Aluminum	EPA 6010	Metals	NELAP	4/6/2004
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	3/4/2008
Ammonia as N	SM 4500-NH3 G	General Chemistry	NELAP	3/4/2008
Aniline	EPA 8270	Extractable Organics	NELAP	10/5/2006
Anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Antimony	EPA 200.7	Metals	NELAP	4/6/2004
Antimony	EPA 6010	Metals	NELAP	4/6/2004
Aramite	EPA 8270	Extractable Organics	NELAP	10/5/2006
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

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E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Arsenic	EPA 200.7	Metals	NELAP	4/6/2004
Arsenic	EPA 6010	Metals	NELAP	4/6/2004
Barium	EPA 200.7	Metals	NELAP	4/6/2004
Barium	EPA 6010	Metals	NELAP	4/6/2004
Benzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Benzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Benzidine	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/5/2006
Beryllium	EPA 200.7	Metals	NELAP	4/6/2004
Beryllium	EPA 6010	Metals	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	3/4/2008
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87646-12, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87646 EPA Lab Code: TN00004 (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/6/2004
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 624	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Cadmium	EPA 200.7	Metals	NELAP	4/6/2004
Cadmium	EPA 6010	Metals	NELAP	4/6/2004
Calcium	EPA 6010	Metals	NELAP	4/6/2004
Carbazole	EPA 8270	Extractable Organics	NELAP	4/6/2004
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Ceriodaphnia dubia	EPA 821-R-02-012 (FW acute)(2002.0)	Toxicity	NELAP	7/1/2009
Ceriodaphnia dubia	EPA 821-R-02-013 (FW chronic)(1002.0)	Toxicity	NELAP	7/1/2009
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	4/6/2004
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chloride	EPA 300.0	General Chemistry	NELAP	4/6/2004
Chlorobenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chlorobenzilate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Chloroethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Chloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 624	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 8260	Volatile Organics	NELAP	4/6/2004

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 200.7	Metals	NELAP	4/6/2004
Chromium	EPA 6010	Metals	NELAP	4/6/2004
Chromium VI	EPA 7196	General Chemistry	NELAP	4/6/2004
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/6/2004
Chrysene	EPA 625	Extractable Organics	NELAP	4/6/2004
Chrysene	EPA 8270	Extractable Organics	NELAP	4/6/2004
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Cobalt	EPA 200.7	Metals	NELAP	4/6/2004
Cobalt	EPA 6010	Metals	NELAP	4/6/2004
Conductivity	EPA 120.1	General Chemistry	NELAP	4/6/2004
Copper	EPA 200.7	Metals	NELAP	4/6/2004
Copper	EPA 6010	Metals	NELAP	4/6/2004
Cyprinella leedsi	EPA 821-R-02-012 (FW acute)(2000.0)	Toxicity	NELAP	7/1/2009
Cyprinodon variegatus	EPA 821-R-02-012 (SW acute)(2004.0)	Toxicity	NELAP	7/1/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Daphnia magna	EPA 821-R-02-012 (FW acute)(2021.0)	Toxicity	NELAP	7/1/2009
Daphnia pulex	EPA 821-R-02-012 (FW acute)(2021.0)	Toxicity	NELAP	7/1/2009
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/6/2004
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dimethoate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/6/2004
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Diphenylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Ethylbenzene	EPA 624	Volatile Organics	NELAP	4/6/2004
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/6/2004
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 625	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluoride	EPA 300.0	General Chemistry	NELAP	4/6/2004

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EPA Lab Code: TN00004

(615) 345-1115

E87646

Empirical Laboratories, LLC.

621 Mainstream Drive

Suite 270

Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	4/6/2004
Hardness (calc.)	EPA 200.7	General Chemistry	NELAP	4/6/2004
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/6/2004
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Ignitability	EPA 1010	General Chemistry	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Iron	EPA 200.7	Metals	NELAP	4/6/2004
Iron	EPA 6010	Metals	NELAP	4/6/2004
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Isophorone	EPA 625	Extractable Organics	NELAP	4/6/2004
Isophorone	EPA 8270	Extractable Organics	NELAP	4/6/2004
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Isosafrole	EPA 8270	Extractable Organics	NELAP	10/5/2006
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006

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Empirical Laboratories, LLC.
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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/6/2004
Lead	EPA 200.7	Metals	NELAP	4/6/2004
Lead	EPA 6010	Metals	NELAP	4/6/2004
Magnesium	EPA 200.7	Metals	NELAP	4/6/2004
Magnesium	EPA 6010	Metals	NELAP	4/6/2004
Manganese	EPA 200.7	Metals	NELAP	4/6/2004
Manganese	EPA 6010	Metals	NELAP	4/6/2004
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Menidia beryllina	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	7/1/2009
Menidia menidia	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	7/1/2009
Menidia peninsulae	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	7/1/2009
Mercury	EPA 245.1	Metals	NELAP	4/6/2004
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methapyrilene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	4/6/2004
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Methyl parathion (Parathion, methyl)	EPA 8270	Extractable Organics	NELAP	10/5/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methylene chloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Methylene chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Molybdenum	EPA 200.7	Metals	NELAP	4/6/2004
Molybdenum	EPA 6010	Metals	NELAP	4/6/2004
Mysidopsis bahia	EPA 821-R-02-012 (SW acute)(2007.0)	Toxicity	NELAP	7/1/2009
Naphthalene	EPA 625	Extractable Organics	NELAP	4/6/2004
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nickel	EPA 200.7	Metals	NELAP	4/6/2004
Nickel	EPA 6010	Metals	NELAP	4/6/2004

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/6/2004
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/6/2004
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
o,o,o-Triethyl phosphorothioate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	4/6/2004
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	3/4/2008
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	10/5/2006
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/6/2004
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
pH	EPA 9040	General Chemistry	NELAP	4/6/2004
pH	SM 4500-H+-B	General Chemistry	NELAP	3/4/2008
Phenacetin	EPA 8270	Extractable Organics	NELAP	10/5/2006
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 625	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phosphorus, total	SM 4500-P E	General Chemistry	NELAP	3/4/2008
Pimephales promelas	EPA 821-R-02-012 (FW acute)(2000.0)	Toxicity	NELAP	7/1/2009
Potassium	EPA 6010	Metals	NELAP	4/6/2004
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/5/2006

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Pyrene	EPA 625	Extractable Organics	NELAP	4/6/2004
Pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Pyridine	EPA 8270	Extractable Organics	NELAP	4/6/2004
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	3/4/2008
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	3/4/2008
Residue-total	SM 2540 B	General Chemistry	NELAP	3/4/2008
Saffrole	EPA 8270	Extractable Organics	NELAP	10/5/2006
Selenium	EPA 200.7	Metals	NELAP	4/6/2004
Selenium	EPA 6010	Metals	NELAP	4/6/2004
Silver	EPA 200.7	Metals	NELAP	4/6/2004
Silver	EPA 6010	Metals	NELAP	4/6/2004
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Sodium	EPA 200.7	Metals	NELAP	4/6/2004
Sodium	EPA 6010	Metals	NELAP	4/6/2004
Styrene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Sulfate	EPA 300.0	General Chemistry	NELAP	4/6/2004
Sulfide	SM 4500-S E (18th Ed.)/TITR	General Chemistry	NELAP	3/4/2008
Sulfotep	EPA 8270	Extractable Organics	NELAP	10/5/2006
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	4/6/2004
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Thallium	EPA 200.7	Metals	NELAP	4/6/2004
Thallium	EPA 6010	Metals	NELAP	4/6/2004
Thionazin (Zinophos)	EPA 8270	Extractable Organics	NELAP	10/5/2006
Tin	EPA 200.7	Metals	NELAP	4/6/2004
Tin	EPA 6010	Metals	NELAP	4/6/2004
Toluene	EPA 624	Volatile Organics	NELAP	4/6/2004
Toluene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	4/6/2004
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/6/2004
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/29/2004
Total organic carbon	SM 5310 C	General Chemistry	NELAP	3/4/2008
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87646-12, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87646

EPA Lab Code:

TN00004

(615) 345-1115

E87646

Empirical Laboratories, LLC.

621 Mainstream Drive

Suite 270

Nashville, TN 37228

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total phenolics	EPA 420.1	General Chemistry	NELAP	4/6/2004
Total phenolics	EPA 420.2	General Chemistry	NELAP	4/6/2004
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	4/6/2004
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	4/6/2004
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Turbidity	EPA 180.1	General Chemistry	NELAP	4/6/2004
Vanadium	EPA 200.7	Metals	NELAP	4/6/2004
Vanadium	EPA 6010	Metals	NELAP	4/6/2004
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Vinyl chloride	EPA 624	Volatile Organics	NELAP	4/6/2004
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Zinc	EPA 200.7	Metals	NELAP	4/6/2004
Zinc	EPA 6010	Metals	NELAP	4/6/2004

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State Laboratory ID: E87646 EPA Lab Code: TN00004 (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	10/5/2006
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004

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Expiration Date: 6/30/2011

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State Laboratory ID: E87646 EPA Lab Code: TN00004 (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/5/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	10/5/2006
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Hexanone	EPA 8260	Volatile Organics	NELAP	4/6/2004
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	10/5/2006
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/6/2004
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/5/2006
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	4/6/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	10/5/2006
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004

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EPA Lab Code: TN00004

(615) 345-1115

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Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	4/6/2004
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	4/6/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	10/5/2006
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Acetone	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/5/2006
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	4/6/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aluminum	EPA 6010	Metals	NELAP	4/6/2004
Aniline	EPA 8270	Extractable Organics	NELAP	10/5/2006
Anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Antimony	EPA 6010	Metals	NELAP	4/6/2004
Aramite	EPA 8270	Extractable Organics	NELAP	10/5/2006
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Arsenic	EPA 6010	Metals	NELAP	4/6/2004
Barium	EPA 6010	Metals	NELAP	4/6/2004
Benzene	EPA 8260	Volatile Organics	NELAP	4/6/2004

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E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/5/2006
Beryllium	EPA 6010	Metals	NELAP	4/6/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/6/2004
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/6/2004
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Bromoform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Cadmium	EPA 6010	Metals	NELAP	4/6/2004
Calcium	EPA 6010	Metals	NELAP	4/6/2004
Carbazole	EPA 8270	Extractable Organics	NELAP	4/6/2004
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/6/2004
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Chloride	EPA 9056	General Chemistry	NELAP	4/6/2004
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Chloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chloroform	EPA 8260	Volatile Organics	NELAP	4/6/2004
Chromium	EPA 6010	Metals	NELAP	4/6/2004
Chromium VI	EPA 7196	Metals,General Chemistry	NELAP	4/6/2004
Chrysene	EPA 8270	Extractable Organics	NELAP	4/6/2004
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Cobalt	EPA 6010	Metals	NELAP	4/6/2004
Copper	EPA 6010	Metals	NELAP	4/6/2004
Dafapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004

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Suite 270
Nashville, TN 37228

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/5/2006
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/6/2004
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Extractable Organics	NELAP	10/5/2006
Diphenylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/5/2006
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluorene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Fluoride	EPA 9056	General Chemistry	NELAP	4/6/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	4/6/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87646-12, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87646** EPA Lab Code: **TN00004** (615) 345-1115

E87646
Empirical Laboratories, LLC.
621 Mainstream Drive
Suite 270
Nashville, TN 37228

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachlorobenzene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/6/2004
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Ignitability	EPA 1010	General Chemistry	NELAP	4/6/2004
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Iron	EPA 6010	Metals	NELAP	4/6/2004
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Isophorone	EPA 8270	Extractable Organics	NELAP	4/6/2004
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Isosafrole	EPA 8270	Extractable Organics	NELAP	10/5/2006
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Lead	EPA 6010	Metals	NELAP	4/6/2004
Magnesium	EPA 6010	Metals	NELAP	4/6/2004
Manganese	EPA 6010	Metals	NELAP	4/6/2004
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Mercury	EPA 7471	Metals	NELAP	4/6/2004
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methapyrilene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/5/2006

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Methylene chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Molybdenum	EPA 6010	Metals	NELAP	4/6/2004
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nickel	EPA 6010	Metals	NELAP	4/6/2004
Nitrate	EPA 9056	General Chemistry	NELAP	4/6/2004
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	4/6/2004
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/6/2004
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	10/5/2006
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	4/6/2004
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/5/2006
Pentachloronitrobenzene (Quintozene)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
Perchlorate	EPA 6850	General Chemistry	NELAP	7/1/2009
pH	EPA 9040	General Chemistry	NELAP	4/6/2004
pH	EPA 9045	General Chemistry	NELAP	4/6/2004
Phenacetin	EPA 8270	Extractable Organics	NELAP	10/5/2006
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phenol	EPA 8270	Extractable Organics	NELAP	4/6/2004
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Potassium	EPA 6010	Metals	NELAP	4/6/2004
Promamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/5/2006

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/5/2006
Pyrene	EPA 8270	Extractable Organics	NELAP	4/6/2004
Pyridine	EPA 8270	Extractable Organics	NELAP	4/6/2004
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Safrole	EPA 8270	Extractable Organics	NELAP	10/5/2006
Selenium	EPA 6010	Metals	NELAP	4/6/2004
Silver	EPA 6010	Metals	NELAP	4/6/2004
Sodium	EPA 6010	Metals	NELAP	4/6/2004
Styrene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Sulfate	EPA 9056	General Chemistry	NELAP	4/6/2004
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	4/6/2004
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	4/6/2004
Thallium	EPA 6010	Metals	NELAP	4/6/2004
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/5/2006
Tin	EPA 6010	Metals	NELAP	4/6/2004
Toluene	EPA 8260	Volatile Organics	NELAP	4/6/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	4/6/2004
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/29/2004
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	4/6/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/6/2004
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/6/2004
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	4/6/2004
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/5/2006
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/6/2004
Vanadium	EPA 6010	Metals	NELAP	4/6/2004
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/5/2006
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/6/2004
Xylene (total)	EPA 8260	Volatile Organics	NELAP	4/6/2004
Zinc	EPA 6010	Metals	NELAP	4/6/2004

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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.

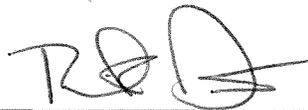
**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

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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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 of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

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 Perken 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 ($<\pm\text{MDL}$ for USACE or $\pm\text{RL}/\text{CRDL}$ for others and CLP), depending on method. **No analyte detected $>2\text{xMDL}$ 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 ($<\pm\text{MDL}$ for USACE or $\pm\text{RL}/\text{CRDL}$ for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected $>2\text{xMDL}$ 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 ($<1/2 \pm\text{RL}$ or $\pm\text{RL}/\text{CRDL}$ for common contaminants (DOD) and $\pm\text{RL}/\text{CRDL}$ 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 ($< \pm MDL$ for USACE or $\pm RL/CRDL$ 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 ($< \pm MDL$ for USACE or $\pm RL/CRDL$ for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected $> 2xMDL$ for DOD.**

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.

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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

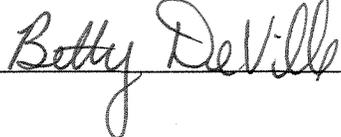
**METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE**

**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 \leq 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 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 302

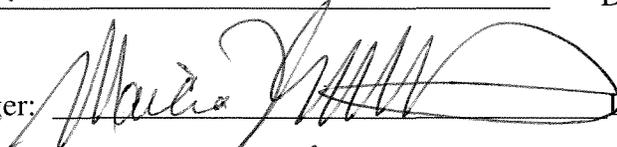
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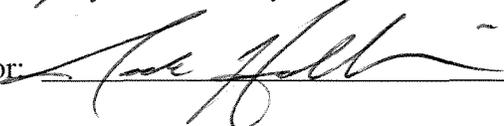
EFFECTIVE DATE: 042610

**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.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
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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

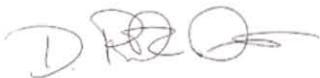
Not applicable to this SOP.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 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.

CFA SOPs and Accreditations



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

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ENVIRONMENTAL

Valid To: May 31, 2012

Certificate Number: 3014.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, 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)) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

High Resolution Gas Chromatography / Mass Spectrometry

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613B	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
2,3,7,8-Tetrachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8-Pentachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
2,3,4,7,8-Pentachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,6,7,8-Heptachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8,9-Heptachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,5,6,7,8-Octachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Tetrachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Pentachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Hexachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Heptachlorodibenzo-p-dioxin	-----	EPA 1613B/8290A	EPA 1613B/8290A

Peter Mlynar

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
Total Tetrachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Pentachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Hexachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
Total Heptachlorodibenzofuran	-----	EPA 1613B/8290A	EPA 1613B/8290A
2-Chlorobiphenyl (1)	-----	EPA 1668A	EPA 1668A
3-Chlorobiphenyl (2)	-----	EPA 1668A	EPA 1668A
4-Chlorobiphenyl (3)	-----	EPA 1668A	EPA 1668A
2,2'-Dichlorobiphenyl (4)	-----	EPA 1668A	EPA 1668A
2,3-Dichlorobiphenyl (5)	-----	EPA 1668A	EPA 1668A
2,3'-Dichlorobiphenyl (6)	-----	EPA 1668A	EPA 1668A
2,4-Dichlorobiphenyl (7)	-----	EPA 1668A	EPA 1668A
2,4'-Dichlorobiphenyl (8)	-----	EPA 1668A	EPA 1668A
2,5-Dichlorobiphenyl (9)	-----	EPA 1668A	EPA 1668A
2,6-Dichlorobiphenyl (10)	-----	EPA 1668A	EPA 1668A
3,3'-Dichlorobiphenyl (11)	-----	EPA 1668A	EPA 1668A
3,4-Dichlorobiphenyl (12)	-----	EPA 1668A	EPA 1668A
3,4'-Dichlorobiphenyl (13)	-----	EPA 1668A	EPA 1668A
3,5-Dichlorobiphenyl (14)	-----	EPA 1668A	EPA 1668A
4,4'-Dichlorobiphenyl (15)	-----	EPA 1668A	EPA 1668A
2,2',3-Trichlorobiphenyl (16)	-----	EPA 1668A	EPA 1668A
2,2',4-Trichlorobiphenyl (17)	-----	EPA 1668A	EPA 1668A
2,2',5-Trichlorobiphenyl (18)	-----	EPA 1668A	EPA 1668A
2,2',6-Trichlorobiphenyl (19)	-----	EPA 1668A	EPA 1668A
2,3,3'-Trichlorobiphenyl (20)	-----	EPA 1668A	EPA 1668A
2,3,4-Trichlorobiphenyl (21)	-----	EPA 1668A	EPA 1668A
2,3,4'-Trichlorobiphenyl (22)	-----	EPA 1668A	EPA 1668A
2,3,5-Trichlorobiphenyl (23)	-----	EPA 1668A	EPA 1668A
2,3,6-Trichlorobiphenyl (24)	-----	EPA 1668A	EPA 1668A
2,3',4-Trichlorobiphenyl (25)	-----	EPA 1668A	EPA 1668A
2,3',5-Trichlorobiphenyl (26)	-----	EPA 1668A	EPA 1668A
2,3',6-Trichlorobiphenyl (27)	-----	EPA 1668A	EPA 1668A
2,4,4'-Trichlorobiphenyl (28)	-----	EPA 1668A	EPA 1668A
2,4,5-Trichlorobiphenyl (29)	-----	EPA 1668A	EPA 1668A
2,4,6-Trichlorobiphenyl (30)	-----	EPA 1668A	EPA 1668A
2,4',5-Trichlorobiphenyl (31)	-----	EPA 1668A	EPA 1668A
2,4',6-Trichlorobiphenyl (32)	-----	EPA 1668A	EPA 1668A
2',3,4-Trichlorobiphenyl (33)	-----	EPA 1668A	EPA 1668A
2',3,5-Trichlorobiphenyl (34)	-----	EPA 1668A	EPA 1668A
3,3',4-Trichlorobiphenyl (35)	-----	EPA 1668A	EPA 1668A
3,3',5-Trichlorobiphenyl (36)	-----	EPA 1668A	EPA 1668A
3,4,4'-Trichlorobiphenyl (37)	-----	EPA 1668A	EPA 1668A
3,4,5-Trichlorobiphenyl (38)	-----	EPA 1668A	EPA 1668A
3,4',5-Trichlorobiphenyl (39)	-----	EPA 1668A	EPA 1668A
2,2',3,3'-Tetrachlorobiphenyl (40)	-----	EPA 1668A	EPA 1668A
2,2',3,4-Tetrachlorobiphenyl (41)	-----	EPA 1668A	EPA 1668A
2,2',3,4'-Tetrachlorobiphenyl (42)	-----	EPA 1668A	EPA 1668A
2,2',3,5-Tetrachlorobiphenyl (43)	-----	EPA 1668A	EPA 1668A
2,2',3,5'-Tetrachlorobiphenyl (44)	-----	EPA 1668A	EPA 1668A
2,2',3,6-Tetrachlorobiphenyl (45)	-----	EPA 1668A	EPA 1668A
2,2',3,6'-Tetrachlorobiphenyl (46)	-----	EPA 1668A	EPA 1668A
2,2',4,4'-Tetrachlorobiphenyl (47)	-----	EPA 1668A	EPA 1668A

Peter Abney

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',4,5-Tetrachlorobiphenyl (48)	-----	EPA 1668A	EPA 1668A
2,2',4,5'-Tetrachlorobiphenyl (49)	-----	EPA 1668A	EPA 1668A
2,2',4,6-Tetrachlorobiphenyl (50)	-----	EPA 1668A	EPA 1668A
2,2',4,6'-Tetrachlorobiphenyl (51)	-----	EPA 1668A	EPA 1668A
2,2',5,5'-Tetrachlorobiphenyl (52)	-----	EPA 1668A	EPA 1668A
2,2',5,6'-Tetrachlorobiphenyl (53)	-----	EPA 1668A	EPA 1668A
2,2',6,6'-Tetrachlorobiphenyl (54)	-----	EPA 1668A	EPA 1668A
2,3,3',4-Tetrachlorobiphenyl (55)	-----	EPA 1668A	EPA 1668A
2,3,3',4'-Tetrachlorobiphenyl (56)	-----	EPA 1668A	EPA 1668A
2,3,3',5-Tetrachlorobiphenyl (57)	-----	EPA 1668A	EPA 1668A
2,3,3',5'-Tetrachlorobiphenyl (58)	-----	EPA 1668A	EPA 1668A
2,3,3',6-Tetrachlorobiphenyl (59)	-----	EPA 1668A	EPA 1668A
2,3,4,4'-Tetrachlorobiphenyl (60)	-----	EPA 1668A	EPA 1668A
2,3,4,5-Tetrachlorobiphenyl (61)	-----	EPA 1668A	EPA 1668A
2,3,4,6-Tetrachlorobiphenyl (62)	-----	EPA 1668A	EPA 1668A
2,3,4',5-Tetrachlorobiphenyl (63)	-----	EPA 1668A	EPA 1668A
2,3,4',6-Tetrachlorobiphenyl (64)	-----	EPA 1668A	EPA 1668A
2,3,5,6-Tetrachlorobiphenyl (65)	-----	EPA 1668A	EPA 1668A
2,3',4,4'-Tetrachlorobiphenyl (66)	-----	EPA 1668A	EPA 1668A
2,3',4,5-Tetrachlorobiphenyl (67)	-----	EPA 1668A	EPA 1668A
2,3',4,5'-Tetrachlorobiphenyl (68)	-----	EPA 1668A	EPA 1668A
2,3',4,6-Tetrachlorobiphenyl (69)	-----	EPA 1668A	EPA 1668A
2,3',4',5-Tetrachlorobiphenyl (70)	-----	EPA 1668A	EPA 1668A
2,3',4',6-Tetrachlorobiphenyl (71)	-----	EPA 1668A	EPA 1668A
2,3',5,5'-Tetrachlorobiphenyl (72)	-----	EPA 1668A	EPA 1668A
2,3',5',6-Tetrachlorobiphenyl (73)	-----	EPA 1668A	EPA 1668A
2,4,4',5-Tetrachlorobiphenyl (74)	-----	EPA 1668A	EPA 1668A
2,4,4',6-Tetrachlorobiphenyl (75)	-----	EPA 1668A	EPA 1668A
2',3,4,5-Tetrachlorobiphenyl (76)	-----	EPA 1668A	EPA 1668A
3,3',4,4'-Tetrachlorobiphenyl (77)	-----	EPA 1668A	EPA 1668A
3,3',4,5-Tetrachlorobiphenyl (78)	-----	EPA 1668A	EPA 1668A
3,3',4,5'-Tetrachlorobiphenyl (79)	-----	EPA 1668A	EPA 1668A
3,3',5,5'-Tetrachlorobiphenyl (80)	-----	EPA 1668A	EPA 1668A
3,4,4',5-Tetrachlorobiphenyl (81)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4-Pentachlorobiphenyl (82)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5-Pentachlorobiphenyl (83)	-----	EPA 1668A	EPA 1668A
2,2',3,3',6-Pentachlorobiphenyl (84)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4'-Pentachlorobiphenyl (85)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5-Pentachlorobiphenyl (86)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5'-Pentachlorobiphenyl (87)	-----	EPA 1668A	EPA 1668A
2,2',3,4,6-Pentachlorobiphenyl (88)	-----	EPA 1668A	EPA 1668A
2,2',3,4,6'-Pentachlorobiphenyl (89)	-----	EPA 1668A	EPA 1668A
2,2',3,4',5-Pentachlorobiphenyl (90)	-----	EPA 1668A	EPA 1668A
2,2',3,4',6-Pentachlorobiphenyl (91)	-----	EPA 1668A	EPA 1668A
2,2',3,5,5'-Pentachlorobiphenyl (92)	-----	EPA 1668A	EPA 1668A
2,2',3,5,6-Pentachlorobiphenyl (93)	-----	EPA 1668A	EPA 1668A
2,2',3,5,6'-Pentachlorobiphenyl (94)	-----	EPA 1668A	EPA 1668A
2,2',3,5',6-Pentachlorobiphenyl (95)	-----	EPA 1668A	EPA 1668A
2,2',3,6,6'-Pentachlorobiphenyl (96)	-----	EPA 1668A	EPA 1668A
2,2',3',4,5-Pentachlorobiphenyl (97)	-----	EPA 1668A	EPA 1668A
2,2',3',4,6-Pentachlorobiphenyl (98)	-----	EPA 1668A	EPA 1668A

Peter Abney

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',4,4',5-Pentachlorobiphenyl (99)	-----	EPA 1668A	EPA 1668A
2,2',4,4',6-Pentachlorobiphenyl (100)	-----	EPA 1668A	EPA 1668A
2,2',4,5,5'-Pentachlorobiphenyl (101)	-----	EPA 1668A	EPA 1668A
2,2',4,5,6'-Pentachlorobiphenyl (102)	-----	EPA 1668A	EPA 1668A
2,2',4,5',6-Pentachlorobiphenyl (103)	-----	EPA 1668A	EPA 1668A
2,2',4,6,6'-Pentachlorobiphenyl (104)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4'-Pentachlorobiphenyl (105)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5-Pentachlorobiphenyl (106)	-----	EPA 1668A	EPA 1668A
2,3,3',4',5-Pentachlorobiphenyl (107)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5'-Pentachlorobiphenyl (108)	-----	EPA 1668A	EPA 1668A
2,3,3',4,6-Pentachlorobiphenyl (109)	-----	EPA 1668A	EPA 1668A
2,3,3',4',6-Pentachlorobiphenyl (110)	-----	EPA 1668A	EPA 1668A
2,3,3',5,5'-Pentachlorobiphenyl (111)	-----	EPA 1668A	EPA 1668A
2,3,3',5,6-Pentachlorobiphenyl (112)	-----	EPA 1668A	EPA 1668A
2,3,3',5',6-Pentachlorobiphenyl (113)	-----	EPA 1668A	EPA 1668A
2,3,4,4',5-Pentachlorobiphenyl (114)	-----	EPA 1668A	EPA 1668A
2,3,4,4',6-Pentachlorobiphenyl (115)	-----	EPA 1668A	EPA 1668A
2,3,4,5,6-Pentachlorobiphenyl (116)	-----	EPA 1668A	EPA 1668A
2,3,4',5,6-Pentachlorobiphenyl (117)	-----	EPA 1668A	EPA 1668A
2,3',4,4',5-Pentachlorobiphenyl (118)	-----	EPA 1668A	EPA 1668A
2,3',4,4',6-Pentachlorobiphenyl (119)	-----	EPA 1668A	EPA 1668A
2,3',4,5,5'-Pentachlorobiphenyl (120)	-----	EPA 1668A	EPA 1668A
2,3',4,5',6-Pentachlorobiphenyl (121)	-----	EPA 1668A	EPA 1668A
2',3,3',4,5-Pentachlorobiphenyl (122)	-----	EPA 1668A	EPA 1668A
2',3,4,4',5-Pentachlorobiphenyl (123)	-----	EPA 1668A	EPA 1668A
2',3,4,5,5'-Pentachlorobiphenyl (124)	-----	EPA 1668A	EPA 1668A
2',3,4,5,6'-Pentachlorobiphenyl (125)	-----	EPA 1668A	EPA 1668A
3,3',4,4',5-Pentachlorobiphenyl (126)	-----	EPA 1668A	EPA 1668A
3,3',4,5,5'-Pentachlorobiphenyl (127)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4'-Hexachlorobiphenyl (128)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5-Hexachlorobiphenyl (129)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5'-Hexachlorobiphenyl (130)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,6-Hexachlorobiphenyl (131)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,6'-Hexachlorobiphenyl (132)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,5'-Hexachlorobiphenyl (133)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,6-Hexachlorobiphenyl (134)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,6'-Hexachlorobiphenyl (135)	-----	EPA 1668A	EPA 1668A
2,2',3,3',6,6'-Hexachlorobiphenyl (136)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5-Hexachlorobiphenyl (137)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5'-Hexachlorobiphenyl (138)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',6-Hexachlorobiphenyl (139)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',6'-Hexachlorobiphenyl (140)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,5'-Hexachlorobiphenyl (141)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,6-Hexachlorobiphenyl (142)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,6'-Hexachlorobiphenyl (143)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5',6-Hexachlorobiphenyl (144)	-----	EPA 1668A	EPA 1668A
2,2',3,4,6,6'-Hexachlorobiphenyl (145)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,5'-Hexachlorobiphenyl (146)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,6-Hexachlorobiphenyl (147)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,6'-Hexachlorobiphenyl (148)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5',6-Hexachlorobiphenyl (149)	-----	EPA 1668A	EPA 1668A

Peter Mlynar

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',3,4',6,6'-Hexachlorobiphenyl (150)	-----	EPA 1668A	EPA 1668A
2,2',3,5,5',6-Hexachlorobiphenyl (151)	-----	EPA 1668A	EPA 1668A
2,2',3,5,6,6'-Hexachlorobiphenyl (152)	-----	EPA 1668A	EPA 1668A
2,2',4,4',5,5'-Hexachlorobiphenyl (153)	-----	EPA 1668A	EPA 1668A
2,2',4,4',5',6-Hexachlorobiphenyl (154)	-----	EPA 1668A	EPA 1668A
2,2',4,4',6,6'-Hexachlorobiphenyl (155)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5-Hexachlorobiphenyl (156)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5'-Hexachlorobiphenyl (157)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',6-Hexachlorobiphenyl (158)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5,5'-Hexachlorobiphenyl (159)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5,6-Hexachlorobiphenyl (160)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5',6-Hexachlorobiphenyl (161)	-----	EPA 1668A	EPA 1668A
2,3,3',4',5,5'-Hexachlorobiphenyl (162)	-----	EPA 1668A	EPA 1668A
2,3,3',4',5,6-Hexachlorobiphenyl (163)	-----	EPA 1668A	EPA 1668A
2,3,3',4',5',6-Hexachlorobiphenyl (164)	-----	EPA 1668A	EPA 1668A
2,3,3',5,5',6-Hexachlorobiphenyl (165)	-----	EPA 1668A	EPA 1668A
2,3,4,4',5,6-Hexachlorobiphenyl (166)	-----	EPA 1668A	EPA 1668A
2,3',4,4',5,5'-Hexachlorobiphenyl (167)	-----	EPA 1668A	EPA 1668A
2,3',4,4',5',6-Hexachlorobiphenyl (168)	-----	EPA 1668A	EPA 1668A
3,3',4,4',5,5'-Hexachlorobiphenyl (169)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5-Heptachlorobiphenyl (170)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',6-Heptachlorobiphenyl (171)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5'-Heptachlorobiphenyl (172)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6-Heptachlorobiphenyl (173)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6'-Heptachlorobiphenyl (174)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5',6-Heptachlorobiphenyl (175)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,6,6'-Heptachlorobiphenyl (176)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4',5,6-Heptachlorobiphenyl (177)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6-Heptachlorobiphenyl (178)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,6,6'-Heptachlorobiphenyl (179)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5,5'-Heptachlorobiphenyl (180)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6-Heptachlorobiphenyl (181)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6'-Heptachlorobiphenyl (182)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5',6-Heptachlorobiphenyl (183)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',6,6'-Heptachlorobiphenyl (184)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,5',6-Heptachlorobiphenyl (185)	-----	EPA 1668A	EPA 1668A
2,2',3,4,5,6,6'-Heptachlorobiphenyl (186)	-----	EPA 1668A	EPA 1668A
2,2',3,4',5,5',6-Heptachlorobiphenyl (187)	-----	EPA 1668A	EPA 1668A
2,2',3,4',5,6,6'-Heptachlorobiphenyl (188)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5'-Heptachlorobiphenyl (189)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5,6-Heptachlorobiphenyl (190)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5',6-Heptachlorobiphenyl (191)	-----	EPA 1668A	EPA 1668A
2,3,3',4,5,5',6-Heptachlorobiphenyl (192)	-----	EPA 1668A	EPA 1668A
2,3,3',4',5,5',6-Heptachlorobiphenyl (193)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (194)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6-Octachlorobiphenyl (195)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (196)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (197)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6-Octachlorobiphenyl (198)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (199)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (200)	-----	EPA 1668A	EPA 1668A

Peter Abney

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (201)	-----	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (202)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5,5',6-Octachlorobiphenyl (203)	-----	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (204)	-----	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5',6-Octachlorobiphenyl (205)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (206)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (207)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (208)	-----	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl (209)	-----	EPA 1668A	EPA 1668A
Soxhlet/Dean-Stark Extraction	-----	-----	EPA 3540C
Continuous Liquid-Liquid Extraction	EPA 3520C	-----	-----





The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

CAPE FEAR ANALYTICAL, LLC

Wilmington, NC

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Presented this 5th day of April 2010.



A handwritten signature in black ink, reading "Peter Abney".

President & CEO
For the Accreditation Council
Certificate Number 3014.01
Valid to May 31, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.



State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E871081

CAPE FEAR ANALYTICAL, LLC
3306 KITTY HAWK ROAD, SUITE 120
WILMINGTON, NC 28405

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - DIOXIN, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, SOLID
AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S

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.

EFFECTIVE December 03, 2010 THROUGH June 30, 2011



A handwritten signature in black ink, appearing to read "Max Salfinger".

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04

NON-TRANSFERABLE E871081-01-12/03/2010
Supersedes all previously issued certificates

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 1 of 17

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081 EPA Lab Code: NC01894 (910) 795-0424

E871081
Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,7,8-TCDD (Dioxin, 2,3,7,8-Tetrachlorodibenzo-p-dioxin)	EPA 1613	Dioxin	NELAP	11/8/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081 EPA Lab Code: NC01894 (910) 795-0424

E871081
Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpdf)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpdf)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpdf)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpdf)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pecdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pecdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pecdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pecdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 194)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ 207)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ 196)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (BZ 197)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ 171)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ 208)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 198)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (BZ 199)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ 172)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 200)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ 201)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 173)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ 174)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5',6-Heptachlorobiphenyl (BZ 175)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ 177)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5-Hexachlorobiphenyl (BZ 129)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ 130)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ 176)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6-Hexachlorobiphenyl (BZ 131)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6'-Hexachlorobiphenyl (BZ 132)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4-Pentachlorobiphenyl (BZ 82)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ 202)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ 178)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ 133)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ 179)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6-Hexachlorobiphenyl (BZ 134)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ 135)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5-Pentachlorobiphenyl (BZ 83)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ 136)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',6-Pentachlorobiphenyl (BZ 84)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3'-Tetrachlorobiphenyl (BZ 40)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ 203)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ 204)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 181)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ 182)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5-Hexachlorobiphenyl (BZ 137)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',6-Hexachlorobiphenyl (BZ 139)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ 140)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4'-Pentachlorobiphenyl (BZ 85)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ 185)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,5'-Hexachlorobiphenyl (BZ 146)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ 186)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ 188)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6-Hexachlorobiphenyl (BZ 142)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ 143)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5',6-Hexachlorobiphenyl (BZ 144)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6-Hexachlorobiphenyl (BZ 147)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ 148)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5',6-Hexachlorobiphenyl (BZ 149)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5-Pentachlorobiphenyl (BZ 86)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5-Pentachlorobiphenyl (BZ 90)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5'-Pentachlorobiphenyl (BZ 97)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ 145)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ 150)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6-Pentachlorobiphenyl (BZ 88)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6'-Pentachlorobiphenyl (BZ 89)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6-Pentachlorobiphenyl (BZ 91)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6'-Pentachlorobiphenyl (BZ 98)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4-Tetrachlorobiphenyl (BZ 41)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4'-Tetrachlorobiphenyl (BZ 42)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,5'-Pentachlorobiphenyl (BZ 92)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ 152)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6-Pentachlorobiphenyl (BZ 93)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6'-Pentachlorobiphenyl (BZ 94)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5',6-Pentachlorobiphenyl (BZ 95)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5-Tetrachlorobiphenyl (BZ 43)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

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Laboratory Scope of Accreditation

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State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,6,6'-Pentachlorobiphenyl (BZ 96)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,6-Tetrachlorobiphenyl (BZ 45)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,6'-Tetrachlorobiphenyl (BZ 46)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3-Trichlorobiphenyl (BZ 16)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ 154)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5-Pentachlorobiphenyl (BZ 99)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ 155)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',6-Pentachlorobiphenyl (BZ 100)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4'-Tetrachlorobiphenyl (BZ 47)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5,6'-Pentachlorobiphenyl (BZ 102)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5',6-Pentachlorobiphenyl (BZ 103)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5-Tetrachlorobiphenyl (BZ 48)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6,6'-Pentachlorobiphenyl (BZ 104)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6-Tetrachlorobiphenyl (BZ 50)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6'-Tetrachlorobiphenyl (BZ 51)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4-Trichlorobiphenyl (BZ 17)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5,6'-Tetrachlorobiphenyl (BZ 53)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',6,6'-Tetrachlorobiphenyl (BZ 54)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',6-Trichlorobiphenyl (BZ 19)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2'-Dichlorobiphenyl (BZ 4)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 205)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ 190)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ 191)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',6-Hexachlorobiphenyl (BZ 158)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ 192)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ 193)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ 159)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 6 of 17

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EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,3',4',5,5'-Hexachlorobiphenyl (BZ 162)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,6-Hexachlorobiphenyl (BZ 160)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5',6-Hexachlorobiphenyl (BZ 161)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5,6-Hexachlorobiphenyl (BZ 163)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5',6-Hexachlorobiphenyl (BZ 164)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5-Pentachlorobiphenyl (BZ 106)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5-Pentachlorobiphenyl (BZ 107)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5'-Pentachlorobiphenyl (BZ 108)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5'-Pentachlorobiphenyl (BZ 122)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,6-Pentachlorobiphenyl (BZ 109)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4-Tetrachlorobiphenyl (BZ 55)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4'-Tetrachlorobiphenyl (BZ 56)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,5',6-Hexachlorobiphenyl (BZ 165)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,5'-Pentachlorobiphenyl (BZ 111)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,6-Pentachlorobiphenyl (BZ 112)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5',6-Pentachlorobiphenyl (BZ 113)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5-Tetrachlorobiphenyl (BZ 57)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5'-Tetrachlorobiphenyl (BZ 58)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',6-Tetrachlorobiphenyl (BZ 59)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3'-Trichlorobiphenyl (BZ 20)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',5,6-Hexachlorobiphenyl (BZ 166)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5',6-Hexachlorobiphenyl (BZ 168)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',6-Pentachlorobiphenyl (BZ 115)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',6-Pentachlorobiphenyl (BZ 119)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4'-Tetrachlorobiphenyl (BZ 60)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5,5'-Pentachlorobiphenyl (BZ 120)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5,5'-Pentachlorobiphenyl (BZ 124)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,5,6-Pentachlorobiphenyl (BZ 116)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4',5,6-Pentachlorobiphenyl (BZ 117)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5',6-Pentachlorobiphenyl (BZ 121)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4',5',6-Pentachlorobiphenyl (BZ 125)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,5-Tetrachlorobiphenyl (BZ 61)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4',5-Tetrachlorobiphenyl (BZ 63)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5-Tetrachlorobiphenyl (BZ 67)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5'-Tetrachlorobiphenyl (BZ 68)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5-Tetrachlorobiphenyl (BZ 70)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5'-Tetrachlorobiphenyl (BZ 76)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,4,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,4,6-Tetrachlorobiphenyl (BZ 62)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4',6-Tetrachlorobiphenyl (BZ 64)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,6-Tetrachlorobiphenyl (BZ 69)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',6-Tetrachlorobiphenyl (BZ 71)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,7,8-Pecdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,4,7,8-Pecdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,4-Trichlorobiphenyl (BZ 21)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4'-Trichlorobiphenyl (BZ 22)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4-Trichlorobiphenyl (BZ 25)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4'-Trichlorobiphenyl (BZ 33)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5,5'-Tetrachlorobiphenyl (BZ 72)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,5,6-Tetrachlorobiphenyl (BZ 65)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5',6-Tetrachlorobiphenyl (BZ 73)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,5-Trichlorobiphenyl (BZ 23)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5-Trichlorobiphenyl (BZ 26)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5'-Trichlorobiphenyl (BZ 34)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,6-Trichlorobiphenyl (BZ 24)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',6-Trichlorobiphenyl (BZ 27)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,7,8-TCDD (Dioxin, 2,3,7,8-Tetrachlorodibenzo-p-dioxin)	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDD (Dioxin, 2,3,7,8-Tetrachlorodibenzo-p-dioxin)	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDF	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDF	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3-Dichlorobiphenyl (BZ 5)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3'-Dichlorobiphenyl (BZ 6)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,4',5-Tetrachlorobiphenyl (BZ 74)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,4',6-Tetrachlorobiphenyl (BZ 75)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,5-Trichlorobiphenyl (BZ 29)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,6-Trichlorobiphenyl (BZ 30)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4',6-Trichlorobiphenyl (BZ 32)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4-Dichlorobiphenyl (BZ 7)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4'-Dichlorobiphenyl (BZ 8)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,5-Dichlorobiphenyl (BZ 9)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,6-Dichlorobiphenyl (BZ 10)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2-Chlorobiphenyl (BZ 1)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5,5'-Pentachlorobiphenyl (BZ 127)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5-Tetrachlorobiphenyl (BZ 78)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5'-Tetrachlorobiphenyl (BZ 79)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4-Trichlorobiphenyl (BZ 35)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',5,5'-Tetrachlorobiphenyl (BZ 80)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',5-Trichlorobiphenyl (BZ 36)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3'-Dichlorobiphenyl (BZ 11)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,4-Trichlorobiphenyl (BZ 37)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,5-Trichlorobiphenyl (BZ 38)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4',5-Trichlorobiphenyl (BZ 39)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4-Dichlorobiphenyl (BZ 12)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4'-Dichlorobiphenyl (BZ 13)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,5-Dichlorobiphenyl (BZ 14)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3-Chlorobiphenyl (BZ 2)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
4,4'-Dichlorobiphenyl (BZ 15)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
4-Chlorobiphenyl (BZ 3)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
Decachlorobiphenyl (BZ 209)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
Total Heptachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010

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Issue Date: 12/3/2010

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 9 of 17

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State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total Hexachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010

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E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzofuran (1,2,3,4,6,7,8-hpcdf)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (1,2,3,4,6,7,8-hpcdd)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8,9-Heptachlorodibenzofuran (1,2,3,4,7,8,9-hpcdf)	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,4,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8,9-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pcdd	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pcdd	EPA 8290	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
1,2,3,7,8-Pcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 194)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (BZ 207)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (BZ 196)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (BZ 197)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Laboratory Scope of Accreditation

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State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC

3306 Kitty Hawk Road, Suite 120

Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',6-Heptachlorobiphenyl (BZ 171)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (BZ 208)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 198)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5',6-Octachlorobiphenyl (BZ 199)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,5'-Heptachlorobiphenyl (BZ 172)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 200)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (BZ 201)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 173)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ 174)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6-Heptachlorobiphenyl (BZ 175)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ 177)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5-Hexachlorobiphenyl (BZ 129)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,5'-Hexachlorobiphenyl (BZ 130)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6,6'-Heptachlorobiphenyl (BZ 176)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6-Hexachlorobiphenyl (BZ 131)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4,6'-Hexachlorobiphenyl (BZ 132)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',4-Pentachlorobiphenyl (BZ 82)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (BZ 202)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5',6-Heptachlorobiphenyl (BZ 178)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,5'-Hexachlorobiphenyl (BZ 133)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6,6'-Heptachlorobiphenyl (BZ 179)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6-Hexachlorobiphenyl (BZ 134)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5,6'-Hexachlorobiphenyl (BZ 135)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',5-Pentachlorobiphenyl (BZ 83)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',6,6'-Hexachlorobiphenyl (BZ 136)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3',6-Pentachlorobiphenyl (BZ 84)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,3'-Tetrachlorobiphenyl (BZ 40)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ 203)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (BZ 204)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 181)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6'-Heptachlorobiphenyl (BZ 182)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 183)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5-Hexachlorobiphenyl (BZ 137)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

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Charlie Crist
Governor



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State Surgeon General

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E871081

Cape Fear Analytical, LLC
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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',6-Hexachlorobiphenyl (BZ 139)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4',6'-Hexachlorobiphenyl (BZ 140)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,4'-Pentachlorobiphenyl (BZ 85)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,5',6-Heptachlorobiphenyl (BZ 185)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,5'-Hexachlorobiphenyl (BZ 146)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6,6'-Heptachlorobiphenyl (BZ 186)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6,6'-Heptachlorobiphenyl (BZ 188)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6-Hexachlorobiphenyl (BZ 142)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5,6'-Hexachlorobiphenyl (BZ 143)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5',6-Hexachlorobiphenyl (BZ 144)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6-Hexachlorobiphenyl (BZ 147)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5,6'-Hexachlorobiphenyl (BZ 148)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5',6-Hexachlorobiphenyl (BZ 149)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5-Pentachlorobiphenyl (BZ 86)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5-Pentachlorobiphenyl (BZ 90)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',5'-Pentachlorobiphenyl (BZ 97)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6,6'-Hexachlorobiphenyl (BZ 145)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6,6'-Hexachlorobiphenyl (BZ 150)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6-Pentachlorobiphenyl (BZ 88)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4,6'-Pentachlorobiphenyl (BZ 89)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6-Pentachlorobiphenyl (BZ 91)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4',6'-Pentachlorobiphenyl (BZ 98)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4-Tetrachlorobiphenyl (BZ 41)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,4'-Tetrachlorobiphenyl (BZ 42)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,5'-Pentachlorobiphenyl (BZ 92)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6,6'-Hexachlorobiphenyl (BZ 152)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6-Pentachlorobiphenyl (BZ 93)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5,6'-Pentachlorobiphenyl (BZ 94)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5',6-Pentachlorobiphenyl (BZ 95)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5-Tetrachlorobiphenyl (BZ 43)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,6,6'-Pentachlorobiphenyl (BZ 96)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,6-Tetrachlorobiphenyl (BZ 45)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3,6'-Tetrachlorobiphenyl (BZ 46)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',3-Trichlorobiphenyl (BZ 16)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5,6'-Hexachlorobiphenyl (BZ 154)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',5-Pentachlorobiphenyl (BZ 99)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',6,6'-Hexachlorobiphenyl (BZ 155)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4',6-Pentachlorobiphenyl (BZ 100)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,4'-Tetrachlorobiphenyl (BZ 47)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5,6'-Pentachlorobiphenyl (BZ 102)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5',6-Pentachlorobiphenyl (BZ 103)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5-Tetrachlorobiphenyl (BZ 48)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6,6'-Pentachlorobiphenyl (BZ 104)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6-Tetrachlorobiphenyl (BZ 50)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4,6'-Tetrachlorobiphenyl (BZ 51)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',4-Trichlorobiphenyl (BZ 17)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5,6'-Tetrachlorobiphenyl (BZ 53)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',6,6'-Tetrachlorobiphenyl (BZ 54)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2',6-Trichlorobiphenyl (BZ 19)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,2'-Dichlorobiphenyl (BZ 4)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,5',6-Octachlorobiphenyl (BZ 205)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5,6-Heptachlorobiphenyl (BZ 190)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5',6-Heptachlorobiphenyl (BZ 191)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4',6-Hexachlorobiphenyl (BZ 158)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,5',6-Heptachlorobiphenyl (BZ 192)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5,5',6-Heptachlorobiphenyl (BZ 193)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,5'-Hexachlorobiphenyl (BZ 159)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

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Laboratory Scope of Accreditation

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State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3,3',4',5,5'-Hexachlorobiphenyl (BZ 162)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5,6-Hexachlorobiphenyl (BZ 160)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5',6-Hexachlorobiphenyl (BZ 161)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5,6-Hexachlorobiphenyl (BZ 163)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5',6-Hexachlorobiphenyl (BZ 164)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5-Pentachlorobiphenyl (BZ 106)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5-Pentachlorobiphenyl (BZ 107)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,5'-Pentachlorobiphenyl (BZ 108)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',5'-Pentachlorobiphenyl (BZ 122)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4,6-Pentachlorobiphenyl (BZ 109)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4-Tetrachlorobiphenyl (BZ 55)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',4'-Tetrachlorobiphenyl (BZ 56)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,5',6-Hexachlorobiphenyl (BZ 165)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,5'-Pentachlorobiphenyl (BZ 111)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5,6-Pentachlorobiphenyl (BZ 112)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5',6-Pentachlorobiphenyl (BZ 113)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5-Tetrachlorobiphenyl (BZ 57)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',5'-Tetrachlorobiphenyl (BZ 58)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3',6-Tetrachlorobiphenyl (BZ 59)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,3'-Trichlorobiphenyl (BZ 20)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',5,6-Hexachlorobiphenyl (BZ 166)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5',6-Hexachlorobiphenyl (BZ 168)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4',6-Pentachlorobiphenyl (BZ 115)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4',6-Pentachlorobiphenyl (BZ 119)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,4'-Tetrachlorobiphenyl (BZ 60)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5,5'-Pentachlorobiphenyl (BZ 120)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5,5'-Pentachlorobiphenyl (BZ 124)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,5,6-Pentachlorobiphenyl (BZ 116)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4',5,6-Pentachlorobiphenyl (BZ 117)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5',6-Pentachlorobiphenyl (BZ 121)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 15 of 17

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4',5',6-Pentachlorobiphenyl (BZ 125)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,5-Tetrachlorobiphenyl (BZ 61)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,5-Tetrachlorobiphenyl (BZ 63)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5-Tetrachlorobiphenyl (BZ 67)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,5'-Tetrachlorobiphenyl (BZ 68)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5-Tetrachlorobiphenyl (BZ 70)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',5'-Tetrachlorobiphenyl (BZ 76)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,6,7,8-Hxcdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,4,6,7,8-Hxcdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,4,6-Tetrachlorobiphenyl (BZ 62)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4',6-Tetrachlorobiphenyl (BZ 64)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4,6-Tetrachlorobiphenyl (BZ 69)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4',6-Tetrachlorobiphenyl (BZ 71)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4,7,8-Pecdf	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,4,7,8-Pecdf	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,4-Trichlorobiphenyl (BZ 21)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,4'-Trichlorobiphenyl (BZ 22)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4-Trichlorobiphenyl (BZ 25)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',4'-Trichlorobiphenyl (BZ 33)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5,5'-Tetrachlorobiphenyl (BZ 72)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,5,6-Tetrachlorobiphenyl (BZ 65)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5',6-Tetrachlorobiphenyl (BZ 73)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,5-Trichlorobiphenyl (BZ 23)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5-Trichlorobiphenyl (BZ 26)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',5'-Trichlorobiphenyl (BZ 34)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,6-Trichlorobiphenyl (BZ 24)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3',6-Trichlorobiphenyl (BZ 27)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3,7,8-TCDD (Dioxin, 2,3,7,8-Tetrachlorodibenzo-p-dioxin)	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDD (Dioxin, 2,3,7,8-Tetrachlorodibenzo-p-dioxin)	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDF	EPA 1613	Extractable Organics	NELAP	11/8/2010
2,3,7,8-TCDF	EPA 8290	Extractable Organics	NELAP	11/8/2010
2,3-Dichlorobiphenyl (BZ 5)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,3'-Dichlorobiphenyl (BZ 6)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,4',5-Tetrachlorobiphenyl (BZ 74)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,4',6-Tetrachlorobiphenyl (BZ 75)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010

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Matrix: Solid and Chemical Materials

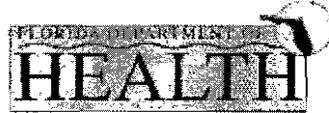
Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,5-Trichlorobiphenyl (BZ 29)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4,6-Trichlorobiphenyl (BZ 30)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4',6-Trichlorobiphenyl (BZ 32)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4-Dichlorobiphenyl (BZ 7)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,4'-Dichlorobiphenyl (BZ 8)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,5-Dichlorobiphenyl (BZ 9)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2,6-Dichlorobiphenyl (BZ 10)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
2-Chlorobiphenyl (BZ 1)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5,5'-Pentachlorobiphenyl (BZ 127)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5-Tetrachlorobiphenyl (BZ 78)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4,5'-Tetrachlorobiphenyl (BZ 79)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',4-Trichlorobiphenyl (BZ 35)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',5,5'-Tetrachlorobiphenyl (BZ 80)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3',5-Trichlorobiphenyl (BZ 36)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,3'-Dichlorobiphenyl (BZ 11)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,4'-Trichlorobiphenyl (BZ 37)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4,5-Trichlorobiphenyl (BZ 38)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4',5-Trichlorobiphenyl (BZ 39)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4-Dichlorobiphenyl (BZ 12)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,4'-Dichlorobiphenyl (BZ 13)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3,5-Dichlorobiphenyl (BZ 14)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
3-Chlorobiphenyl (BZ 2)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
4,4'-Dichlorobiphenyl (BZ 15)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
4-Chlorobiphenyl (BZ 3)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
Decachlorobiphenyl (BZ 209)	EPA 1668	Pesticides-Herbicides-PCB's	NELAP	11/8/2010
Total Heptachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Heptachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 12/3/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 17 of 17

Attachment to Certificate #: E871081-01, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871081

EPA Lab Code: NC01894

(910) 795-0424

E871081

Cape Fear Analytical, LLC
3306 Kitty Hawk Road, Suite 120
Wilmington, NC 28405

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total Hexachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Hexachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Pentachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzofuran	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzofuran	EPA 8290	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzo-p-dioxin	EPA 1613	Extractable Organics	NELAP	11/8/2010
Total Tetrachlorodibenzo-p-dioxin	EPA 8290	Extractable Organics	NELAP	11/8/2010

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 12/3/2010

Expiration Date: 6/30/2011

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

**CAPE FEAR ANALYTICAL, LLC
LABORATORY WASTE MANAGEMENT PLAN**

DOCUMENT NUMBER CF-LB-G-001

(REVISION 0)

PROPRIETARY INFORMATION

This document contains proprietary information that is the exclusive property of Cape Fear Analytical, LLC (CFA). No contents of this document may be reproduced or otherwise used for the benefit of others except by express written permission of CFA.

Cape Fear Analytical, LLC (CFA) Laboratory Waste Management Plan has been prepared in accordance with Local, State, and Federal Laws. This plan, which is part of CFA's corporate standard operations and quality systems, specifies the management of laboratory wastes by CFA.

Approved by:

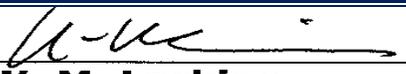
CFA	<p>CFA's Document Control Officer certifies this document to be a true copy of the fully executed original.</p> <p> W. M. Larkins</p>
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1. INTRODUCTION

This plan addresses the management of laboratory wastes generated by Cape Fear Analytical, LLC (CFA) at the facility located at 3306 Kitty Hawk Road, Suite 120 in Wilmington, North Carolina. It is designed to ensure that laboratory wastes are handled and disposed in compliance with all applicable Local, State and Federal regulations in a manner that minimizes impact to the environment and potential liability to CFA and our clients.

The State of North Carolina currently classifies CFA as a Small Quantity Generator. CFA abides by the criteria set forth in North Carolina Hazardous Waste Management Regulations. In compliance with this, CFA does not:

1. Generate more than 1,000 kilograms of hazardous waste in a calendar month and may only accumulate hazardous waste onsite for 180 days or less.
2. Accumulate onsite more than 6,000 kilograms of hazardous waste.
3. Accumulate onsite more than 1.0 kilograms of acutely hazardous waste.

CFA maintains a current record of the monthly hazardous waste accumulation totals in order to facilitate compliance with state regulations. CFA also maintains a monthly inspection log of Satellite Accumulation Areas.

If, for any reason, it is determined that regulation criteria are not being met or are in danger of not being met, the laboratory management must be notified immediately.

Samples and other materials used in analytical procedures do not become waste until all analytical and contract archive requirements have been met.

All wastes are grouped into one of several waste management categories according to the following characterization criteria:

- Chemical, radiochemical, and physical characterization
- Potential or known hazardous characteristics
- Applicable regulatory requirements
- Appropriate recycle options
- Disposal methods
- Client specific instructions

These waste management categories, a description of the wastes, and the management procedures are summarized in Table 1.

Any materials that do not fall into a designated Waste Category should be brought to the attention of the Laboratory Director for evaluation.

2. DISCUSSION

This document details the procedures for the identification, collection, handling and disposal of excess client samples as well as laboratory wastes. It is intended for use by laboratory personnel before, during, and after sample analysis. The purpose of this plan is to ensure that the receipt, handling, discharges and shipments from this facility are in compliance with applicable environmental regulations. In addition, this SOP contains the corporate RCRA Emergency/Contingency Plan for laboratory and environmental emergencies. (See Appendix 1.)

- 2.1 CFA's waste management plan is designed to ensure the health and safety of laboratory personnel and the environment. The plan is implemented to:
 - 2.1.1 Minimize the volume of samples and waste generated, and
 - 2.1.2 Recycle, reclaim, and reuse whenever it is possible to do so.

2.2 Waste Identification and Communication

- 2.2.1 Project Evaluation - Waste identification begins during Contracts and Proposal evaluations with the receipt of a Request for Proposal (RFP). Based on client supplied site information, any known or suspected hazards are identified, lab-handling precautions are evaluated and disposal options for the material are determined prior to bidding on projects. If a proposal is submitted and subsequently awarded to CFA, this information is provided to the appropriate CFA personnel for project setup, prior to the receipt of samples.
- 2.2.2 Client Knowledge - During the receipt of sample material, the laboratory must identify packages shipped as "Hazardous Materials". This identification process is a useful tool for applying a client's generator knowledge to the material received so that appropriate handling and segregation can occur within the laboratory. Clients may identify known hazards including applicable EPA hazardous waste codes in the receiving paperwork or chain of custody.
- 2.2.3 Internal Communication – Client-identified hazards are entered into AlphaLIMS indicating the hazardous constituent. This hazardous indication is additionally communicated via the sample container barcode label. Hazardous Constituents discovered by the laboratory during the analysis of samples are additionally available in the AlphaLIMS system. The hazardous indication is carried with the sample through laboratory handling and disposal.
- 2.2.4 Laboratory Knowledge - Analytical testing performed at CFA generates a wide variety of laboratory-derived wastes. Each analytical procedure is profiled by applying process knowledge to determine the chemical characteristics of the resulting derived wastes produced during the analysis. This profile plays a key role in determining the appropriate handling, storage, segregation, and disposal of the resulting waste.
- 2.2.5 Waste Segregation - During sample analysis, analysts shall segregate the resulting laboratory-derived wastes into the appropriate Satellite Collection Container. This segregation is determined by considering the matrix of the material being analyzed, the established waste profile for the analysis being performed, and any hazardous constituents identified in the sample during the receipt process. Should the analyst have any questions as to the appropriate Satellite Collection Container, they should contact a member of the laboratory management. When a Satellite Collection Container is determined to be full, the material must be collected and transferred to the Sample Archive and Waste Management Area.
- 2.3 Sample Handling - Active client samples are held in the areas specified in CF-SR-E-001 Sample Receipt, Login and Storage. When contract or regulatory retention requirements are met, samples are moved from these storage areas and transferred to the Sample Archive and Waste Management Area to be archived until contractual requirements authorize disposal. Once samples are approved for disposal in LIMS, samples are scanned for disposal in accordance to SOP CF-LB-E-012.
- 2.4 Sample Exemption - Material sent for analysis is exempt from hazardous waste regulations during the time period it is considered a sample. This time period includes collection, analysis, while being held after analysis, and while the sample is being returned to the client. Client samples not being returned to the client, where CFA is responsible for the disposal may be composited with similar matrix samples to facilitate disposal. It is at this point that the material loses its exemption as a sample, and is considered a waste material. Analytical testing and

generator knowledge will be utilized to determine the correct disposal disposition of the laboratory waste in accordance with applicable regulations.

3. DEFINITIONS

The following definitions are used in this plan.

- 3.1 **AlphaLIMS** - CFA's laboratory information management system
- 3.2 **Characteristic Hazardous Waste** - Wastes exhibiting one or more of the following:
1. Ignitability (D001) - This category includes any liquid with a flashpoint less than 140°F, or non-liquids capable of producing fire due to friction or moisture, or ignitable compressed gases.
 2. Corrosivity (D002) - This category includes material that has a pH less than or equal to 2 or greater than or equal to 12.5, or able to corrode steel at a rate greater than 0.25 inches per year.
 3. Reactivity (D003) - This category includes materials that are normally unstable and readily undergo violent change without detonation, or react violently with water, or form potentially explosive mixtures with water, or generate toxic gases when mixed with water, or are either Class A, Class B, or forbidden explosives.
 4. Toxicity (D004-D043) - This category includes material in which the extract from a waste contains any of the contaminants listed in Table 3 at or above the concentration listed in the table.
- 3.3 **EPA RCRA Empty** - Empty containers are defined as having been emptied by conventional means (pumping, pouring, and aspirating) and containing less than 1" of residue and/or <3% of the container volume (whichever is less). For acute hazardous (**P-listed**) wastes, containers must be triple rinsed with an appropriate solvent or the intact inner liner of the container removed for the container to be considered **empty**. The rinsate and liner must be disposed of as hazardous waste.
- 3.4 **Hazardous Constituent** - Any substance that has been defined as hazardous waste in regulations promulgated by the Administrator of the United States Environmental Protection Agency.
- 3.5 **Hazardous Material** - According to the US DOT, a hazardous material is defined as "A substance or material, which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated".
- 3.6 **Hazardous Waste** - Discarded materials, including solid, liquid, semisolid or contained gaseous material, resulting from operations or from activities which because of its quantity, concentration or physical, chemical or infectious characteristics may cause or significantly contribute to an increase in mortality or an increase in serious irreversible or incapacitating reversible illness or pose a substantial present or potential hazard to human health or the environment if improperly treated, stored, transported, disposed of or otherwise managed.
- 3.7 **Hazardous Waste Satellite Collection Container** - Containers designated for the accumulation of hazardous waste "in containers at or near any point of generation where wastes initially accumulate, provided it is under control of the operator of the process generating the waste". The containers must be marked with the words "Hazardous Waste" or other words that identify the waste. Hazardous Satellite Collection Containers must be closed unless waste is being added or removed from the container, the container must be in good condition, and must be compatible with the waste being added. Regulations allow a maximum of 55 gallons of hazardous waste or 1 quart of acutely hazardous waste for each separate waste stream being

- accumulated under satellite conditions. However, the waste streams should be distinguishable either by waste code, specific TSD facility handling requirements, or other criteria, which justifies handling in separate containers. Full containers of hazardous waste may be stored at the point of generation for a maximum of 3 days before being transferred to a designated, main accumulation area.
- 3.8 **Laboratory Derived Waste** - Discarded material generated during the analytical procedure.
- 3.9 **Listed Hazardous Waste** - The EPA maintains four lists of wastes specified in 40 Parts 261.30-35 they are summarized below as:
1. **"F" Listed** wastes from non-specific sources.
 2. **"P" Listed** on-specification and off-specification wastes, which are commercial chemical products or manufacturing chemical intermediate, which are acutely, hazardous wastes based on toxicity and reactivity.
 3. **"U" Listed** on-specification and off-specification wastes which are commercial chemical products or manufacturing chemical intermediates based on toxicity.
 4. **"K" Listed** wastes from specific sources.
- 3.10 **Material Requiring Special Handling** - These materials do not fall within a normal waste management category at CFA, or they have been identified as material requiring "Special Instruction" for handling.
- 3.11 **Non Hazardous Waste** - Waste that does not meet the definition of Hazardous Waste.
- 3.12 **PCB Hazardous Waste** - Any substance that is no longer of use and is limited to the biphenyl molecule that has been chlorinated to >50 PPM or any combination of substances which contains such substance at >50 PPM.
- 3.13 **Sample Archive and Waste Management Area** - Sample Archives is the area where Client Samples are held pending disposal or return to the client. Client Samples are transferred from the areas specified in CF-SR-E-001, Sample Receipt, Login and Storage, to Sample Archive when all analytical procedures have been completed and regulatory holding times have expired. The Client Samples remain in this area pending contractual sample retention periods. After this time, the samples are prepared for disposal or return to the client. The Waste Management Area is where appropriate storage containers of Hazardous, Non-hazardous, & Mixed Material is classified/profiled and accumulated pending disposal. Expired Chemicals and Laboratory Standards are also lab packed in this area.
- 3.14 **Satellite Collection Container** - These are containers located throughout the laboratory for the collection of various matrices of: Non Hazardous Waste, Hazardous Waste, RCRA empty sample and reagent containers, and Laboratory Sanitary Trash.
- 3.15 **Solid waste** - Any garbage, refuse, sludge and other discarded material, including solid, liquid, semisolid, or contained gaseous material.
- 3.16 **Treatment** - A method, technique or process designed to change the physical, chemical or biological character or composition of hazardous waste so as to neutralize such waste or to render such waste non-hazardous, safer for transport, amenable for recovery, amenable for storage or reduced in volume.
- 3.17 **TSDF** - A facility that is permitted and licensed to Treat, Store, and Dispose waste material.
- 3.18 **Universal Waste Regulation** - Hazardous waste managed via a set of regulations providing management standards in lieu of the regulations specified under 40 CFR parts 260 through 272. Universal Waste Regulations include: (A) Batteries as described in 40 CFR 273.2;(B) Pesticides as described in 40 CFR273.3; (C) Thermostats as described in 273.4; and (D) Lamps

as described in 40 CFR 273.5.

- 3.19 **Waste Acceptance Criteria (WAC)** - Acceptance criteria specified by a TSDF for Characterizing, Packaging, and Documenting waste prior to authorizing the receipt of waste from a Generator. Established to comply with the Local, State and Federal regulations.
- 3.20 **Foreign Soil** - USDA regulated soil from foreign sources, including Hawaii, Guam, Puerto Rico, and the US Virgin Islands, and some domestic soil originating in areas currently quarantined by the USDA-APHIS, PPQ Office.
- 3.21 **Instrument Process Collection Container** - Containers utilized for immediate and temporary collection of hazardous and non-hazardous effluents integral to any process associated with an instrument. This may include containers directly receiving effluents from flow-through type instruments or for vial storage following instrument unloading. This material will enter a waste stream when removed from the instrument and is determined to have no analytical value.

4. SAFETY AND SECURITY

- 4.1 All personnel should exercise proper safety precautions when handling any waste material. Gloves, aprons, and safety glasses must be used when handling hazardous materials.
- 4.2 Hazardous Satellite Collection Containers **must** remain closed except when they are being filled or emptied.
- 4.3 "Ignitable/Flammable Waste" Hazardous Satellite Collection Containers require additional safety measures that may include: bonding and grounding assemblies, flame arrestors, and special storage in flammable cabinets or fume hoods rated for use with ignitables/flammables.
- 4.4 Hazardous Satellite Collection Containers and Hazardous Waste Accumulation Areas shall be inspected weekly for leaks or signs of adverse chemical reactions. Any indications of leaks or chemical reactions shall be documented and corrected as soon as possible.

5. WASTE DISPOSAL

WARNING

DO NOT DISPOSE OF CHEMICALS, SAMPLES OR LABORATORY DERIVED WASTE RESIDUES UNTIL YOU ARE SURE OF THE PROPER WASTE MANAGEMENT PROCEDURE. CONTACT THE LABORATORY DIRECTOR OR QUALITY MANAGER IF ADDITIONAL GUIDANCE IS NEEDED.

- 5.1 Laboratory waste is collected and accumulated in appropriately labeled Satellite Collection Containers. These containers are located throughout the laboratory in Sample Collection and Accumulation Areas. To comply with regulations and ensure that material is handled safely and properly, "once full" containers of hazardous waste may only be stored at the point of generation for a maximum of 3 normal business days before being transferred to a designated, main accumulation area.
- 5.2 Hazardous constituents known to be contained within samples are communicated to the laboratory via the AlphaLIMS system. This information can include applicable EPA waste codes, PCBs, Dioxin, Foreign Soil and other known or suspected hazardous constituents. The information communicated via the barcode label, along with knowledge of the analytical process being performed determines the appropriate management of the waste material
- 5.3 **Material Requiring Special Handling** - These materials do not fall within a normal and/or routine waste management category at CFA, or they have been identified as

material requiring "Special Instruction" for handling. Indication for material of this nature may be included in AlphaLIMS or a project kick off meeting.

- 5.4 Non-Hazardous / Non Radioactive - These wastes are both Non Hazardous and Non Radioactive. This determination is made utilizing client provided information, analytical results, and generator knowledge of processes creating the wastes (Table 1, categories 2, 4, 8, 10 summarize material within this category).
- 5.5 Reusable/Recyclable Solvents - This material may be beneficially reused by the laboratory or other industries. This practice minimizes the volume of Hazardous Waste Generated by the Laboratory (Table 1, category 1 summarizes material within this category).
- 5.6 RCRA Hazardous / Non Radioactive - These Wastes are both Hazardous and Non Radioactive. This determination is made utilizing client provided information, analytical results, and generator knowledge of processes creating the wastes (Table 1, category 6 summarizes material within this category).
- 5.7 Universal Waste – This waste consists of discarded items such as batteries, mercury containing thermostats, and mercury containing lamps.

6. SANITARY SEWER DISCHARGE

A permit is not required to discharge wastewater from the laboratory facility to the Sanitary Sewer.

7. HISTORY

Revision 0 – Initiated SOP,

TABLE 1: SUMMARY OF WASTE MANAGEMENT CATEGORIES

Category Name	Waste Streams	Management
1 Recyclable Solvents	Material that may fall within this category: <ul style="list-style-type: none"> Methylene Chloride 95% Water 5% 	<ul style="list-style-type: none"> Accumulate in approved Containers Accumulate derived wastes in SCCs(1) Beneficially Reuse Material Handled by TSDF.
2 Flammable Mixed Solvents	Material that falls within this waste category: <ul style="list-style-type: none"> Methanol 10-20% Water 10-20% Toluene 10-30% Hexane 10-20% Dichloromethane <15% Other Solvents 30-60% 	<ul style="list-style-type: none"> Accumulate in approved Containers Accumulate derived wastes in SCCs(1) Handled by TSDF.
3 Waste Soil, Sludge, Solids, and Oils with Dioxin	Material that falls within this waste category: <ul style="list-style-type: none"> Excess Soil, Sludge, Solids, and Oils Soil 85-90% Plastic/Glass debris 10-15% Dioxins <0.5% 	<ul style="list-style-type: none"> Samples remain in original containers pending contractual archive period, at which time they may be composited with like material to facilitate disposal Dispose by incineration at approved Hazardous TSDF
4 Wastewater with Dioxin	Material that falls within this waste category: <ul style="list-style-type: none"> Excess Water Water 99-100% Dioxins <0.5% 	<ul style="list-style-type: none"> Samples remain in original containers pending contractual archive period, at which time they may be composited with like material to facilitate disposal Dispose by incineration at approved Hazardous TSDF
6 Laboratory trash	Material that falls within this waste category: <ul style="list-style-type: none"> Non Hazardous consumable laboratory supplies generated during handling/analysis of samples 	<ul style="list-style-type: none"> Non Hazardous Only Disposed in sanitary landfill

(1) SCC = Satellite Collection Container

TABLE 2: PROCEDURE FOR WASTE DISPOSAL TO AN APPROVED TSDF

1. Based on generator knowledge or analytical testing, the waste is assigned to a CFA approved waste stream.
2. The waste is then matched with a CFA approved TSDF using the applicable “Waste Acceptance Criteria” for that facility. Approved waste Brokers may be used in the selection process.
3. A waste profile is then generated and submitted, or verified on file with the selected TSDF.
4. A signed contract, approved purchase order, or other binding correspondence must be in place to secure acceptable pricing. If not, the approved Broker or the TSDF shall be contacted to obtain a quote for disposal cost.
5. Specific administrative and pre-transportation requirements vary with each TSDF. It is the responsibility of CFA’s Waste Management personnel to ensure that the specific TSDF’s policies and procedures are followed.
6. Upon approval of the waste profile by the TSDF, all documentation shall be generated for the shipment in accordance with state of North Carolina regulations. This may include the following:
 - a. Hazardous Waste Manifests
 - b. Non-Hazardous Waste Manifests
 - c. Land Disposal Restriction Notifications (as applicable)
 - d. Shipping Labels
7. Also upon approval of the waste profile by the TSDF, transportation arrangements shall be made for pick-up, transport, and delivery of the waste material.
8. All material to be shipped and the required documentation shall be ready on the day of pick-up. The conveyance and all material must be inspected prior to loading. Driver credentials must be verified as well. Once loaded, a CFA trained shipper shall ensure the material is properly secured, incompatible material is segregated, and the vehicle is properly placarded.
9. Prior to departure, all manifests must be signed by both a CFA trained shipper and the driver. Proper documentation is provided to both parties and any special instructions provided to the driver.
10. All appropriate shipment information is then entered into the Laboratory Information System (LIMS) and copies of appropriate documents are scanned for electronic record retention.
 - 10.1 For hazardous waste, a signed copy of the manifest should be received from the TSDF within 35 days of shipment receipt. If a signed manifest is not received in that time frame, the transporter/TSDF shall be contacted to determine the status of the shipment.

TABLE 3: HAZARDOUS WASTE LIMITS FOR THE TOXICITY CHARACTERISTIC

	Contaminant	Regulatory Limit (ppm)
D019	Carbon Tetrachloride	0.5
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D023	o-Cresol	200.0
D024	m-Cresol	200.0
D025	p-Cresol	200.0
D026	Cresol	200.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13
D032	Hexachlorobenzene	0.13
D033	Hexachlorobutadiene	0.5
D034	Hexachloroethane	3.0
D035	Methyl Ethyl Ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0
D039	Tetrachloroethylene	0.7
D040	Trichloroethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D043	Vinyl Chloride	0.2

FEDERAL, STATE & LOCAL EMERGENCY RESPONSE CONTACTS

ORGANIZATION	CONTACT & LOCATION	PHONE
LOCAL		
Primary Fire Dept.		911
Secondary Fire Dept.		911
Emergency Response Team	Under Fire Department hazardous material team	911
Sheriff's Dept.		911
Local State Police		910-395-3917
Hospital		910-343-7000
STATE		
State Police HQ		911
State Environmental HQ		919-733-2423
State Emergency Response		911
FEDERAL		
Nearest EPA Office	EPA Region IV - Atlanta	404-874-0607
Nearest Coast Guard Office	US Coast Guard, Wilmington	910-772-2200
	U.S. Coast Guard	Chemtrec
	National Response Center	Emergency Response
	800-424-8802	800-424-9300
		Poison Center
		800-922-1117

Arrangements with local police, fire departments, hospitals and state emergency response services:

A copy of the contingency plan has been provided to the County Sheriff's Department, fire department, hospitals and the local office of the North Carolina Department of Health and the Environment. Letters of acceptance were provided for them to respond that they had received the plan.

PRIMARY EMERGENCY COORDINATOR, CHRIS CORNWELL**FUNCTIONS/ACTIVITIES DURING EMERGENCY**

Primary emergency coordinator will be responsible for assessment and control in the event of a fire, explosion or unplanned release of hazardous waste or hazardous waste constituents to the air, soil, or surface waters at this facility. The coordinator would provide the resources necessary to contain released materials, ensure the safety of employees and the public, and contact the proper emergency response officials.

SECONDARY EMERGENCY COORDINATOR MIKE LARKINS**FUNCTIONS/ACTIVITIES DURING EMERGENCY**

Assist primary coordinator. In the event the primary coordinator is unavailable, the secondary coordinator would assume primary responsibilities for the event.

Facility Location**Facility #**

3306 Kitty Hawk Rd, Suite 120, Wilmington, NC 28405

Facility Description: The facility is a specialty environmental laboratory. The laboratory building has approximately 5,000 square foot of floor space for the laboratory equipment and office space. The building has code-locked controlled access. Fire and security protection is provided on a 24-hour basis. Fire detectors and pull alarms are located throughout the facility. Some areas have temperature alarms to notify the 24-hour security service in the event of high temperature conditions not related to a fire. Facility diagrams are placed strategically in the facility.

Entrances The main entrance is identified on the facility diagram. The entrance to the waste management area is accessible to authorized personnel.	First Aid Stations Basic first aid stations for cuts & minor burns are located in various areas of the lab.	Hazardous Materials Storage Area Identified on the facility diagram.
Emergency Exits Exits are identified on the facility diagram.	Communication System/Speakers Phones are located throughout the facility. A phone is located in the waste management area.	Waste Storage Area Identified on the facility diagram.
Fire Extinguishing System Individual fire extinguishers and automatic halon systems are identified on the facility diagram.	Evacuation Routes Evacuation routes and exits are posted though out the laboratory.	Decontamination Equipment Decontamination would involve use of the same materials used for spill control listed above.
Spill Control Equipment Absorbent, lime, granular carbon, spill booms, shovels and brooms are located in the waste management area. Closed trenches in the waste management area floor would provide containment in the event of a spill.	Eye Wash Stations and Emergency Showers An eyewash station is located in the waste management area. Additional eye and full body wash stations are identified on the facility diagram.	Fire Systems/Alarms Fire detectors are located throughout the facility. In the event an alarm is activated, a fire alarm sounds and a signal is sent to the security company.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

DIOXIN/FURAN/PCB CONGENER SAMPLE PROCESSING

(CF-OA-E-001 REVISION 3)

APPLICABLE TO METHODS:
EPA SW-846 Method 8290A, EPA Method 1613B and EPA Method 1668B

PROPRIETARY INFORMATION

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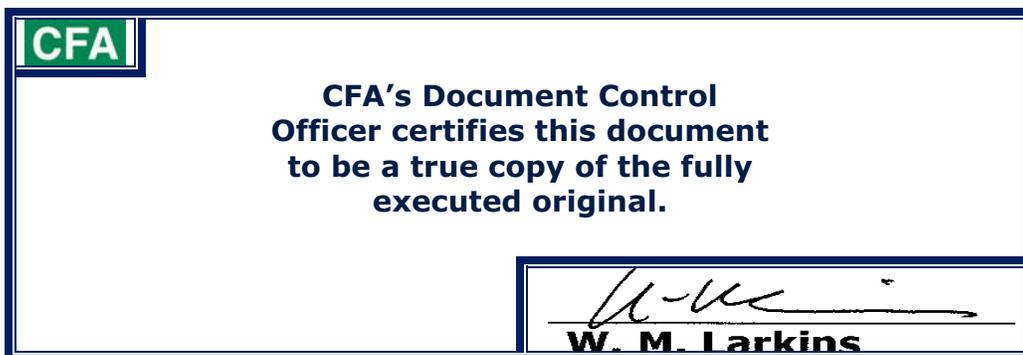


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1.0 STANDARD OPERATING PROCEDURE FOR DIOXIN/FURAN/PCB CONGENER SAMPLE PROCESSING

2.0 METHOD OBJECTIVE, PURPOSE, CODES, AND SUMMARY

- 2.1 This standard operating procedure provides the necessary instructions for the preparation, extraction and cleanup of environmental samples for analysis by high-resolution GC/MS methods listed below, for the determination of polychlorinated dibenzo-dioxins/furans(PCDDs/PCDFs) and polychlorinated biphenyls (PCBs).
- 2.2 Solid and tissue samples are homogenized as needed and extracted by soxhlet/Dean-Stark (SDS). Aqueous samples are extracted by continuous liquid-liquid extraction (CLLE). Sample extracts are fractionated and prepared for analysis using a variety of cleanup procedures, including silica gel and florisil column chromatography.
- 2.3 Analytical methods
 - 2.3.1 EPA SW-846 Method 8290A
 - 2.3.2 EPA Method 1613B
 - 2.3.3 EPA Method 1668B
- 2.4 Extraction techniques
 - 2.4.1 Method 3520C (CLLE)
 - 2.4.2 Method 3540C (SDS)
- 2.5 Cleanup techniques
 - 2.5.1 Method 3620C (Florisil)
 - 2.5.2 Method 3630C (Silica)

3.0 APPLICABLE MATRICES

This SOP is applicable to solid (soil, sediment, sludge), aqueous (groundwater, surface water, leachate, drinking water) and tissue matrices.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

- 4.1 Calibration ranges and PQLs may be found in the appropriate analytical SOPs.
- 4.2 Analysts must demonstrate proficiency prior to work under this SOP. Demonstrated proficiency may be in the form of an IDOC or PT study. Records are maintained in the Quality Department.

5.0 METHOD VARIATIONS

- 5.1 Aqueous samples containing >1% solids are filtered prior to extraction. The aqueous portion is extracted by CLLE, and the filter and solids are extracted by SDS. The fractions are re-combined prior to cleanup.
- 5.2 Aqueous samples are adjusted to a pH of ≤ 7 using 50:50 sulfuric acid. Samples with an initial pH >10 are re-checked after acidifying to ensure a pH of ≤ 7 .
- 5.3 EPA Method 3520C, Continuous Liquid-Liquid Extraction and EPA Method 3540C, Soxhlet Extraction include the use and control of laboratory equipment not in use at Cape Fear Analytical, LLC. The laboratory SOP correctly reflects the equipment used to perform the preparatory methods of reference.

6.0 DEFINITIONS

- 6.1 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.

- 6.2 **Blank:** An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.
- 6.3 **Cleanup Standards:** Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1613B uses one compound (37Cl4-2378-TCDD) as the Cleanup Standard. Method 1668B uses three compounds as cleanup standards. Method 8290A does not address the use of cleanup standards.
- 6.4 **Extraction Standards:** Isotopes added prior to extraction that serve as internal standards. In addition, to measure the overall extraction and fractionation efficiencies.
- 6.5 **Laboratory Control Standard/Duplicate (LCS/LCSD):** Aliquots of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS/LCSD are analyzed exactly like a sample, and their purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 6.6 **Laboratory Duplicate (DUP):** Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The duplicate aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.
- 6.7 **Matrix Spike and Matrix Spike Duplicate (MS and MSD):** Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.

7.0 INTERFERENCES/LIMITATIONS

- 7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.
- 7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.
- 7.3 Reagents and solvents should be purified and tested before use, or alternatively, they should be purchased pre-cleaned by the manufacturer.
- 7.4 PCB extracts must not be allowed to concentrate to dryness due to the potential for loss of low molecular weight chlorinated biphenyls.

8.0 SAFETY PRECAUTIONS AND WARNINGS

WARNING

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

NO OCCUPATIONAL EXPOSURE LIMIT FOR DIOXIN HAS BEEN ESTABLISHED. IT IS A KNOWN AND PROBABLE HUMAN CARCINOGEN.

PCBs HAVE BEEN TENTATIVELY CLASSIFIED AS KNOWN OR SUSPECTED HUMAN OR MAMMALIAN CARCINOGENS.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS FROM SOLIDS.

- 8.1 Eye protection should be worn when handling samples, reagents, or standards.
NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.
- 8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.
- 8.3 Personal Protective Equipment (PPE)
 - 8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.
 - 8.3.2 Analysts should prepare samples and standards under the hood.
- 8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.
- 8.5 Never leave gas cylinders unchained or untied.
- 8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.
- 8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.
- 8.8 For further safety instructions, consult the Safety, Health and Chemical Hygiene Plan, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

- 9.1 Equipment associated with this SOP includes:
 - Soxhlet/Continuous Liquid-Liquid Extraction Rack
 - Soxhlet/Dean-Stark (SDS) glassware apparatus
 - Continuous Liquid-Liquid (CLLE) glassware apparatus
 - Balances
 - Fume hoods
 - Turbovap
 - Dry weight oven
 - Buchner funnels

- Vacuum pump
 - 2 L vacuum flask
- 9.2 Materials and supplies:
- Boiling chips
 - Cellulose thimbles
 - Spatulas
 - Aluminum dry weight pans
 - pH strips
 - 5 3/4" disposable pipets and rubber pipet bulbs
 - Filter paper
 - Nitrogen
 - 60 mL vials with PTFE caps
 - 60 mL vial racks
 - 25 mL drying columns
 - 10 mL drying columns
 - Glass wool
 - 10-100 µL air displacement pipet and disposable tips
 - 100-1000 µL air displacement pipet and disposable tips
 - 1000 mL graduated cylinder

10.0 REAGENTS AND STANDARDS

10.1 Reagents, chemicals, and standards:

- Methylene chloride
- Toluene
- Hexane
- Carbon—Carbopack C or equivalent
- Celite
- Tridecane: Plug a 25 mL drying column with glass wool; add 1 g sodium sulfate; add 2 g carbon-coated celite. Percolate the tridecane from the supplier's bottle through the carbon column. Collect into a clean and properly labeled container; document preparation in the Supply Prep logbook.
- Nonane
- Deionized water
- Sodium sulfate
- Concentrated sulfuric acid
- 1 N Sodium hydroxide
- High purity potassium hydroxide

- Neutral silica
- Acid-coated silica: 44.0 g concentrated sulfuric acid to 100 g neutral silica. Add 1 to 2 mL acid to the silica and shake vigorously; continue until all acid has been added. Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- NaOH-coated silica: 30.0 g 1 N NaOH to 100 g neutral silica. Add 1 to 2 mL base to the silica and shake vigorously; continue until all base has been added. Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- KOH-coated silica: Dissolve 56 g high purity KOH in 300 mL methanol. Add 100 grams neutral silica; stir on hot plate at 60° to 70°C for 1 to 2 hours. Decant liquid and rinse with two 100 mL aliquots methanol. Rinse once with 100 mL methylene chloride. Spread potassium silicate on foil and dry for 1 to 2 hours in a fume hood. Activate at 200 °C for 18 hours. Stir in 37 g neutral silica (This is a deviation from the methods, intended to reduce the strength of the KOH-coated silica to prevent recovery losses in the samples). Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- Florisil
- Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These include extraction standard, matrix spiking standard, cleanup standard, injection standard. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference CF-LB-E-007 for further information regarding standards and their preparation.

11.0 SAMPLE HANDLING AND PRESERVATION

- 11.1 For method 8290A, samples must be extracted within 30 days of collection. For methods 1613B and 1668B, samples must be extracted within 1 year of collection. Sample extracts have a 45-day holding time from the date of extraction by method 8290A and a 365 day holding time from the date of extraction by 1613B and 1668B. Note that per method 8290A, tissue extracts must be completely analyzed by 45 days from collection.
- 11.2 Samples should be collected in amber glass containers with PTFE-lined caps.
- 11.3 Aqueous samples should be checked in the field for the presence of residual chlorine, and if present, preserved with 80 mg sodium thiosulfate. If sample labels do not indicate whether they have been checked/preserved, the samples should be checked at the time of receipt, and preserved if necessary.
- 11.4 Samples should be maintained at $0 \leq 6$ °C after collection until such time as they may be disposed by the laboratory.
- 11.5 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to extract into their custody.
- 11.6 All samples and sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

- 12.1 Sample preparation and conditioning

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12.1.1 Homogenization of tissue

- Vegetation samples can require special procedures to prevent cross contamination from other matrices. Some vegetation can be difficult to reduce in particle size and homogenize. Vegetation sample processing is handled on a case-by-case basis with project management to client communication.
- Tissue samples should be received and maintained at -10 to -20°C. If a sample is received as a whole specimen (fish, rodent, etc.), a project manager should verify with the client which portion of the specimen is to be analyzed.
- If specific portions must be dissected from the whole specimen, the remaining tissue can be refrozen. When the entire sample, usually fish, requires analysis, reduce the sample down to manageable means using a butcher knife.

NOTE: Great care must be exercised when reducing a frozen fish with a knife. Cut-resistant gloves must be worn to protect the hands as well as to hold the fish in place.

- When analyzing specific portions, such as a fillet, dissect the required portion under stringent safety guidelines to prevent injury as well as to ensure the integrity of the sample.
- Once reduced in size or dissected to the appropriate portion, process the tissue through a meat grinder, if required, and collect into an amber glass jar.
- To ensure a homogeneous mixture, grind the sample two more times.
- Record any observations on the bench sheet.

12.1.2 Compositing

Specific procedures for compositing may be found in SOP CF-LB-E-031.

12.1.3 Percent solids determination

Specific procedures for determination of percent solids (moisture content) may be found in SOP CF-OA-E-020.

12.1.4 Percent lipids determination

- Extract the sample by soxhlet using methylene chloride. Do not add tridecane.
- Concentrate the extract to approximately 20 mL and transfer to a tared 60 mL vial.
- Concentrate the extract to dryness in a Turbovap.
- Allow to cool; take an initial weight using an analytical balance.
- Store the extract vial in a dessicator for 12 hours and re-weigh. Continue to perform re-weighs every hour until constant weight is achieved (<1% change).
- Calculate percent lipids as follows:

$$\%L = \frac{R}{W} \times 100$$

Where:

R = weight of dry residue in grams

W = initial weight of sample in grams

12.1.6 Sub-Sampling

Sample matrices and mechanisms of contamination are infinitely variable and require judgments to be made. If the client provides no instructions, it is advisable that experienced analysts decide which sub-sampling techniques are employed. For solid samples, mixing is the preferred process. For liquid samples without obvious layers, the entire container is used and rinsed. Documentation of these two forms of sub-sampling is not required due to the frequency of their use.

All other selected sub-sampling techniques should be documented. If the sample integrity or composition does not match that anticipated by the laboratory, the client must be contacted to confirm or clarify any sub-sampling instructions. An example of a time when client direction is required can be demonstrated with the receipt of a coring sleeve. If upon opening the container, the analyst notices obvious heterogeneous composition of the sample, (clay in one end of the sleeve, and sand at the other end), appropriate instructions from the client must be obtained prior to beginning analysis.

Anytime the analytical result will obviously be biased, it must be documented, and the client should be notified prior to beginning any sub-sampling technique. An example of a time when this occurs is when interferences or target contamination require the use of a reduced extraction volume for a liquid sample preventing a container rinse.

12.2 Sample Extraction

12.2.1 Soxhlet/Dean-Stark (SDS) Extraction

12.2.1.1 Pre-extraction

- Fill 500 mL round-bottom flask with 350 mL extraction solvent (methylene chloride for all tissues and PCB solids; toluene for dioxin/furan solids).
- Add one scoop of boiling chips to the round-bottom and attach soxhlet and Dean-Stark glassware. If pre-extracting for PCB samples, also include the thimble. If samples to be extracted are not extremely wet (i.e. >30% moisture), the Dean-Stark may not be needed.
- Clamp the apparatus securely to the extraction rack and attach the condenser.
- Pull out emergency stops to energize heater controllers.
- Set the heating mantle controller to '65' for toluene or '50' for methylene chloride, turn power switch to 'on', and allow to reflux for 3 to 4 hours.
- Turn power switch to 'off' and allow apparatus to cool.
- Hit emergency stops to turn off controllers.
- Disassemble condenser and SDS and dump solvent into appropriate waste container.
- Glassware is now prepared for extraction.

12.2.1.2 Extraction of Solid Samples

- Refill the round-bottom flask with 350 mL extraction solvent.
- Add one scoop fresh boiling chips.
- For dioxin/furan extractions, add 500 uL tridecane as a keeper solvent. Do NOT add tridecane for PCB extractions.
- Reassemble the glassware apparatus, clamp securely to the extraction rack.
- Weigh a 10 g dry weight equivalent soil aliquot into a cellulose thimble.
- Add 10 to 15 g sodium sulfate to the thimble and mix thoroughly.
- Place the thimble into the appropriately labeled soxhlet.
- Add the appropriate type and amount of extraction standard (spike) to the thimble (see Appendix 1).
- Attach a Dean-Stark adaptor (if needed) and attach the condenser.
- Allow the sample 1 – 2 hours for equilibration
- Pull out emergency stops.
- Set the heating mantle controller to ‘65’ for toluene or ‘50’ for methylene chloride, turn power switch to ‘on’, wrap the round-bottom flask with aluminum foil and allow to reflux for 18 to 24 hours.
- At the end of the extraction period, open the stopcock on the soxhlet and allow the solvent to drain to waste.
- When the solvent level reaches 30 to 40 mL, turn off the heating mantle controller and allow the apparatus to cool.
- Hit emergency stops.
- Remove the condenser, Dean-Stark and soxhlet from the round-bottom flask, and place the cellulose thimble into the appropriate waste can.
- Quantitatively transfer the extract from the round-bottom flask to an appropriately labeled 60 mL vial.
- Concentrate the extract under nitrogen in a Turbovap. Dioxin/furan extracts should be concentrated to 500 µL (tridecane) and exchanged with hexane. PCB extracts should be concentrated to approximately 2 to 3 mL to avoid analyte loss. DO NOT TAKE PCB EXTRACTS TO DRYNESS.
- Add the appropriate type and amount of cleanup standard to the extract (see Appendix 1).
- The extract is now ready for cleanup.

12.2.2 Aqueous Extraction

12.2.2.1 Continuous Liquid-Liquid Pre-extraction

- Verify water level in circulation tank.
- Turn the two-way valves to ‘extract’ and turn on the pump switch to ‘extract’. Verify water is flowing.
- Turn on the water heater to 160 °F.

- Add 1 to 2 boiling chips to the concentrator, fill the drying adapter halfway with sodium sulfate, and assemble the glassware apparatus.
- Securely attach the apparatus to the CLLE extraction rack. Attach the water hoses to the concentrator.
- Add 200 mL methylene chloride to the extractor body. Then add 1 L deionized water, being careful not to allow any water into the sidearm.
- Attach the condenser to the apparatus.
- Verify chiller temperature and flow.
- Open the water valves to allow hot water to flow through the concentrator.
- Open the sidearm stopcock to allow methylene chloride to flow over to the drying adapter.
- Allow pre-extraction to run for 3 to 4 hours.
- Close stopcock and allow methylene chloride to concentrate to 15-20 mL.
- Turn off heater, close water valves, turn two-way valves to 'drain', and turn pump switch to 'drain'.
- Once the water has drained, turn the pump switch to 'off'.
- Remove the concentrator and drying adapter and pour remaining methylene chloride into the appropriate waste container, leaving the boiling chips inside.
- Rinse twice more with methylene chloride and dump to waste.
- Re-attach the drying adapter.
- Remove the extractor body and dump the deionized water and methylene chloride in the appropriate waste container.
- Re-attach the entire apparatus to the extraction rack.
- Glassware is now ready for sample extraction.

12.2.2.2 High- solids (>1%) Procedure

- Remove water sample from the cooler and allow to come to room temperature.
- Pipet 1-2 mL of sample onto a pH strip and record pH on bench sheet.
- Spike the sample with the appropriate type and amount of extraction standard (see Appendix 1)
- Allow sample to equilibrate for 1 – 2 hours
- Place sample on balance and tare.
- Attach large Buchner funnel to 2L vacuum flask.
- Attach vacuum pump hose to vacuum flask.
- Place sheet of glass fiber filter paper inside funnel and rinse with toluene.
- Pull off toluene with vacuum and discard.

- Pour sample into funnel and apply vacuum.
 - Reweigh the sample bottle on the tared scale and record the resulting sample volume on the bench sheet.
 - Rinse the sample bottle with 50 mL deionized water and add to funnel.
 - Continue to pull vacuum until all water has been pulled through the filter.
 - Turn off vacuum pump.
 - Remove filter paper and place in soxhlet extractor. Extract using procedure in Section 12.2.1.
 - Pour water into extractor body and extract using procedure in Section 12.2.2.3. NOTE: SAMPLE HAS ALREADY BEEN SPIKED. DO NOT RESPIKE.
- 12.2.2.3 Low-solids (<1%) Extraction by Continuous Liquid-Liquid (CLLE)
- Fill extractor body with 250 mL methylene chloride.
 - Remove water sample from the cooler and allow to come to room temperature.
 - Pipet 1 to 2 mL of sample onto a pH strip and record pH on bench sheet.
 - Place sample on balance and tare.
 - Shake to thoroughly homogenize the sample, and pour into the appropriately labeled extractor body, being sure the sample does not get into the sidearm.
 - Reweigh the sample bottle on the tared scale and record the resulting sample volume on the bench sheet.
 - Rinse the sample bottle with 50 mL methylene chloride and pour into the extractor body.
 - Spike the sample with the appropriate type and amount of extraction standard (see Appendix 1).
 - Allow the sample to equilibrate for 1 – 2 hours.
 - Add 2 mL 50:50 sulfuric acid to the extractor body. If initial pH was >10, re-check pH; if still > 7, continue adding acid to achieve a pH <= 7.
 - Attach the condenser.
 - Turn the two-way valves to ‘extract’ and turn on the pump switch to ‘extract’.
 - Turn on the water heater.
 - Open the water valves.
 - Open the stopcock.
 - Allow the sample to extract for 18 to 24 hours.
 - Close the stopcock and allow the extract to concentrate down to the nipple of the concentrator. If samples are for PCB analysis, only concentrate to 5 to 10 mL to avoid analyte loss.

- Turn heater off, close water valves, switch two-way valves to 'drain' and turn pump to 'drain'.
- When water has drained, turn pump to 'off'.
- Remove concentrator and drying adapter.
- Pour contents of extractor body into the appropriate waste container.
- Remove drying adapter from concentrator and dump sodium sulfate into appropriate waste container.
- Quantitatively transfer the extract to an appropriately labeled 60 mL vial.
- Add the appropriate type and amount of cleanup standard to the vial. For dioxin/furan extracts, add 100 μ L tridecane to the vial.
- Concentrate the extracts down to the 100 μ L tridecane in the Turbovap at 45 °C. For PCB extracts, only concentrate down to 2 to 3 mL.
- Remove the vial and cap.
- Extract is now ready for cleanup.

12.2.3 Cleanup Procedures

12.2.3.1 Acid/Base Silica Column (for dioxin/furan samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column from bottom to top as follows: 1 g neutral silica gel, 4 g basic silica gel, 1 g neutral silica gel, 8 g acidic silica gel, 2 g neutral silica gel, 4 g sodium sulfate.
- Rinse the column with 50 mL hexane and catch in a waste container.
- Position a 60 mL vial under the column.
- Quantitatively transfer the extract onto the column.
- Elute the sample with 50 mL hexane.
- Remove the 60 mL vial and cap; position a new 60 mL vial under the column.
- Elute with an additional 50 mL hexane.
- Concentrate the two vials to approximately 5 ml in the Turbovap at 45 °C and combine into one vial.
- If the extract requires additional cleanup, concentrate to approximately 1 to 2 mL and proceed to another cleanup step.
- If the extract is ready for analysis, concentrate to dryness and add 0.5 mL methylene chloride.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.2 Acid/Base Silica Column (for PCB samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column from bottom to top as follows: 1 g neutral silica gel, 4 g basic silica gel, 1 g neutral silica gel, 8 g acidic silica gel, 2 g neutral silica gel, 4 g sodium sulfate.
- Rinse the column with 50 mL hexane and catch in a waste container.
- Position a 60 mL vial under the column.
- Quantitatively transfer the extract onto the column.
- Elute the sample with 25 mL hexane.
- If the extract requires additional cleanup, concentrate to approximately 1 to 2 mL in the Turbovap at 45 °C and proceed to another cleanup step.
- If the extract is ready for analysis, concentrate to 0.5 mL. DO NOT let PCB extracts go to dryness.
- Quantitatively transfer the extract to a labeled GC vial containing 18 µL nonane and deliver to the GC/MS analyst.

12.2.3.3 Florisil Column (for dioxin/furan samples)

- Insert a glass wool plug into a 10 mL disposable pipet, pack with 1.5 g florisil, and top with 1 g sodium sulfate.
- Rinse the column with 10 mL methylene chloride, 10 mL 2% methylene chloride/hexane, and 10 mL hexane, and discard to waste.
- Quantitatively transfer the extract to the column with hexane.
- Rinse the column with 10 mL hexane.
- Elute with 20 mL 2% methylene chloride/hexane and discard to waste.
- Position a 60 mL vial under the column and elute the sample with 35 mL methylene chloride.
- Concentrate the extract in the Turbovap at 45 °C to approximately 0.5 mL.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.4 Anthropogenic Isolation Column (for all tissue samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column as follows: 2 g neutral silica gel, 2 g potassium silicate, 2 g sodium sulfate, 10 g acid silica gel, 2 g sodium sulfate.
- Elute the column with 100 mL hexane and collect to a waste container.
- Quantitatively transfer the extract to the column.
- Elute with 200 mL hexane and collect into an appropriate sample container.
- If the sample requires further cleanup, concentrate the extract to approximately 1 to 2 mL hexane in the Turbovap at 45 °C and proceed with another cleanup.

- If no further cleanup is required, concentrate the extract to dryness (for dioxin/furan samples) and add 0.5 mL methylene chloride. For PCB samples, concentrate only to approximately 0.5 mL. DO NOT take PCB samples to dryness.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.5 Acid/Base Back-Extraction (for PCB samples)

- Partition the extract against 50 mL potassium hydroxide solution.
- Shake for 2 minutes, venting periodically.
- Remove and discard the aqueous layer.
- Repeat until no color is visible in the aqueous layer, to a maximum of four washings.
- Partition the extract against 50 mL sodium chloride solution.
- Discard the aqueous layer.
- Partition the extract against 50 mL sulfuric acid.
- Discard the aqueous layer.
- Repeat until no color is visible in the aqueous layer, to a maximum of four washings.
- Partition the extract against 50 mL sodium chloride solution.
- Discard the aqueous layer.
- Transfer the extract to a drying column containing 7-10 grams sodium sulfate.
- Concentrate the extract to 1 to 2 mL hexane in the Turbovap at 45°C and proceed with additional cleanup steps.

13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

13.1 Method Blank (MB)

A method blank is extracted with each extraction batch of 20 samples or less. The method blank should be a reagent-free matrix similar to that of the batch, such as deionized water, glass beads or purified corn oil. The method blank is spiked in the same manner as the samples. The method blank is subjected to the same extraction and cleanup procedures as the samples, and is used as a measure of laboratory contamination. Method blank acceptance criteria may be found in the appropriate analytical SOP.

13.2 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

A laboratory control sample is extracted with each extraction batch of 20 samples or less. The LCS should be a reagent-free matrix (as listed above), and is subjected to the same extraction and cleanup procedures. The LCS is spiked with native analytes in addition to the extraction standards added to all samples. The LCS is subjected to the same extraction and cleanup procedures as the samples, and is used as a measure of system performance. A LCSD may also be performed as required by the analytical method. LCS/LCSD acceptance criteria may be found in the appropriate analytical SOP.

13.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS/MSD pair may be performed as required by the analytical method, or as requested by a specific client. A matrix spike is a second aliquot of sample which is spiked with native analytes prior to extraction, and is used as another measure of system performance. MS/MSD acceptance criteria (if available) may be found in the appropriate analytical SOP.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

Samples are analyzed using a Waters Autospec Premier high-resolution GC/MS system. Instrument calibration, standardization and performance information may be found in the appropriate analytical SOP.

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

Procedures for analysis and instrument operation may be found in the appropriate analytical SOP.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Chiller water levels should be checked monthly.

16.2 Chromalox heater water level should be checked each day of use.

16.3 Balance

Procedures for balance calibration and maintenance may be found in SOP CF-LB-E-002 for Balances.

16.4 Fume Hood

Fume hood monitoring and maintenance may be found in SOP CF-FC-E-003.

16.5 Dry weight oven temperature should be verified each day of use.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data such as sample weights/volumes, dry weights, pH, spike amounts and lot numbers are input to the LIMS.

17.2 Calculations such as percent moisture and percent lipids are calculated in the LIMS.

18.0 POLLUTION/CONTAMINATION

18.1 Work areas should be maintained free of dust and dirt accumulations.

18.2 Whenever possible, work should be performed in a fume hood to reduce the spread of solvent fumes and airborne contaminants.

18.3 Glassware should be washed and prepared in a designated area.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Bench data are input manually to AlphaLIMS. The data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-extraction, the use of a new spike mix, or a more complex set of actions. When troubleshooting measures (Section 21) fail to bring an analytical process or data into control, a nonconformance report (NCR) and/or corrective action should be initiated in accordance with CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when a sample or QC fails to meet defined acceptance criteria. Troubleshooting may involve one or more of the following actions:

- 21.1 When a method blank fails the defined criteria, the analyst must find and eliminate the source of contamination before proceeding with the analysis. This may involve the further testing of reagents, solvents, equipment or glassware. If the contamination is believed to be from a highly contaminated sample in the batch, the sample may be removed from the batch and the remaining batch re-processed.
- 21.2 If an extraction standard recovery fails the specified criteria, the sample must be evaluated as to why the failure may have occurred. If matrix interferences are apparent, the extract may need further cleanup steps, or the sample may need to be re-extracted at a reduced weight/volume.
- 21.3 If any native analyte recoveries in the LCS are outside specified limits, the process should be examined. Corrective action may include repeating the extraction or analysis, utilizing a new spike mix, or more complex actions.
- 21.4 If normal equipment and software operating procedures do not resolve troubleshooting efforts, the manuals for software, hardware and other equipment discussed in this SOP are available for consultation and resolution. On-line support may be available from software and instrument manufacturers, as well. Any revisions, repairs or corrective actions required must be documented in accordance with the laboratory's Quality System as described in CF-QS-B-001.

22.0 RECORDS MANAGEMENT

- 22.1 Bench data logbooks are stored in the lab in storage boxes as long as there is space available. When space runs out, the boxes are sent to off-site storage.
- 22.2 Records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

24.0 REFERENCES

- 24.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 8290A, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry (HRGC/HRMS)," Rev. 1, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.
- 24.2 Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Rev. B, Oct. 1994. USEPA, Office of Water, Engineering and Analysis Division, 401 M Street SW, Washington, DC 20460.
- 24.3 Method 1668B, "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS", November 2008. USEPA, Office of Water, Office of Science and Technology, Engineering and Analysis Division, 1200 Pennsylvania Avenue NW, Washington, DC 20460.

25.0 HISTORY

Revision 0: New document.

Revision 1: Updated solid reference matrix in Section 13. Added Appendix 1, Spike Profiles. Added vegetation sample processing comments in section 12.1.1.

Revision 2: Clarified pH requirements, revised extraction time to match reference method, re-ordered some bulleted steps to match actual practices, changed to nonane as PCB solvent, added method variation section, added discussion of software and equipment troubleshooting support.

Revision 3. Removed reference to SOP CF-LB-E-032 (retired) in Section 12.1.3. Removed references to 8290 cleanup standard.

Appendix 1: Spike Profiles

1613	Volume (μL)	[Conc] (ng/ μL)	Amt. (ng)	Final Vol. (μL)	[Inst] (pg/ μL)	[Sx] (pg/g)
1613ES	40	0.05	2	20	100	200
1613MX _(LCS)	40	0.005	0.2		10	20
1613CS	20	0.01	0.2		10	20
8290JS*	20	0.1	2		100	200

8290	Volume (μL)	[Conc] (ng/ μL)	Amt. (ng)	Final Vol. (μL)	[Inst] (pg/ μL)	[Sx] (pg/g)
8290ES	40	0.05	2	20	100	200
1613MX* _(LCS)	40	0.005	0.2		10	20
8290JS	20	0.1	2		100	200

1668	Volume (μL)	[Conc] (ng/ μL)	Amt. (ng)	Final Vol. (μL)	[Inst] (pg/ μL)	[Sx] (pg/g)
1668ES	40	0.05	2	20	100	200
1668MX _(LCS)	40	0.0125	0.5		25	50
1668CS	40	0.05	2		100	200
1668JS	2	1	2		100	200

* Standard is interchangeable between methods 8290 and 1613

Complete compound lists for each standard may be found in the standards logbook.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE ANALYSIS OF POLYCHLORINATED DIBENZO-p-DIOXINS

AND POLYCHLORINATED DIBENZOFURANS (PCDDs/PCDFs)

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-

RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

(CF-OA-E-002 REVISION 8)

APPLICABLE TO METHODS:

EPA SW-846 Method 8290A, EPA Method 1613B, EPA SW-846 Method 0023A, EPA Method TO-9a

PROPRIETARY INFORMATION

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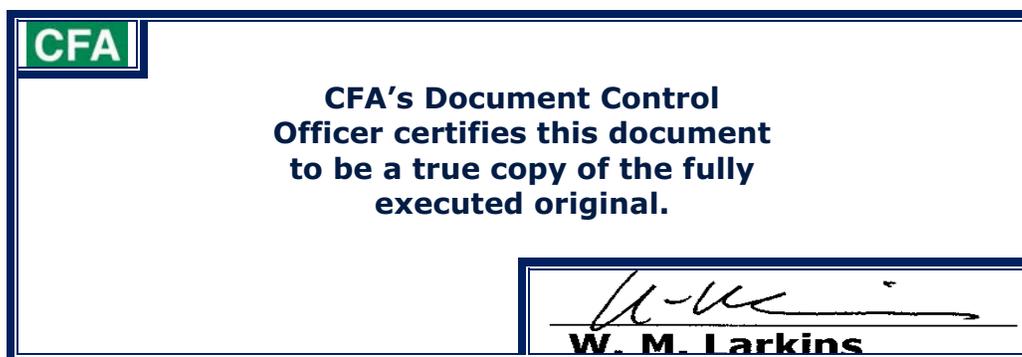


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1.0 STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXINS AND POLYCHLORINATED DIBENZOFURANS (PCDD/PCDF) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

This standard operating procedure (SOP) covers the analytical determination of PCDD/PCDFs according to the following methods:

- 2.1 SW-846 Method 8290A
- 2.2 EPA Method 1613B
- 2.3 SW-846 Method 0023A
- 2.4 EPA Method TO-9a (Jan 99)

3.0 APPLICABLE MATRICES

Applicable matrices for methods 8290A and 1613B include groundwater, wastewater, surface water, leachate, soil, sediment, sludge, oil, and tissue. The applicable matrix for method 0023A is an air sampling train, which may contain XAD resin, filters, impinger water and solvent rinses. TO-9a is an ambient air sampling train which may contain polyurethane foam (PUF), XAD resin, filters, and solvent rinses.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

- 4.1 Methods 8290A, 1613B and 0023A may be used to quantify PCDD/PCDFs that are soluble in methylene chloride and/or toluene. The compounds are separated using a gas chromatograph (GC) and detected using a high-resolution double focusing mass spectrometer (HRMS). Appendix 1 lists the analytes currently analyzed using these methods and their practical quantitation limits.
- 4.2 The practical quantitation limit (PQL) is the lowest level in the calibration curve. The PQL is the lowest level at which compounds may be accurately quantitated and is compound dependent. The calibration curve typically ranges from 1.0 ng/mL to 1000 ng/mL for methods 8290A, 0023A, and TO-9a, and from 0.5 ng/mL to 2000 ng/mL for method 1613B. These ranges reflect instrument readings, which are in ng/mL (ppb). It should be noted that the calibration range may vary between calibrations and instruments.
- 4.3 Method detection limit studies (MDLs) are performed and/or verified on an annual basis. MDLs are done for aqueous, solid, tissue and XAD matrices. For more information regarding MDLs, refer to The Determination of Method Detection Limits, CF-LB-E-001.
- 4.4 Qualified analysts must demonstrate proficiency initially and annually thereafter with an IDOC, CDOC, or PT study. Acceptability criteria may be found in the applicable analytical method.
 - 4.4.1 To establish the ability to generate acceptable accuracy and precision, the analyst should perform an "analyst validation study" or Initial Demonstration of Capability. Four LCS standards are extracted and analyzed. Calculate the average recovery and the standard deviation of the recovery for each analyte of interest using the four results. Then compare the average and the standard deviation with the corresponding criteria found in Table 6 of method 1613B,

or with the determined limits for methods 8290A and 0023A. If the average and the standard deviation for all analytes of interest meet the acceptance criteria, then the analyst may begin work on actual samples. If the validation study fails for one or more of the compounds, then the study must be repeated for those compounds which failed.

5.0 METHOD VARIATIONS

- 5.1 Cape Fear Analytical analyzes a calibration point at 0.25 ng/mL, which is below the method required low point.
- 5.2 Standards and sample extracts are stored at room temperature to avoid analyte loss. Many of the target analytes in these methods form a strong cohesive bond with solids such as glass in cold temperatures; this type of analyte loss is not addressed in the method. (This is a variance from the following method recommendations: $\leq 6^\circ$ per method 8290A; $< -10^\circ\text{C}$ per 1613B; -10° to -20°C per DoD QSM.)
- 5.3 Cape Fear Analytical utilizes the DB-5MS GC column, which is capable of better resolution of the TCDF isomers. This column exhibits a different elution pattern than the DB-5 column referenced in the analytical methods. Relative retention time limits have been determined for this column for use with method 1613B, and are listed in Table 8.
- 5.4 Method 1613B does not address the reporting of EDL and EMPC. These values are reported for this method only when requested by the client.

6.0 DEFINITIONS

- 6.1 Accuracy: The degree of agreement between an observed value and an accepted reference value.
- 6.2 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.
- 6.3 Blank: An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.
- 6.4 Calibration Standard (CAL): An aliquot of a primary standard solution or stock standard solution. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 6.5 Calibration Verification Standard (CVS, CCAL, CS3WT): A solution of target analytes with a concentration near the mid-point of the calibration range. It should be obtained from a second source vendor and is used to verify the initial calibration on a basis described in the determinative method. This solution may also contain the window defining analytes and the column performance mix.
- 6.6 Cleanup Standards: Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1613B uses one compound (37Cl4-2378-TCDD) as the Cleanup Standard. Method 8290A does not address the use of cleanup standards.

- 6.7 Duplicate Analysis: The analysis or measurement of the variable of interest performed identically on two field subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision of sample, preservation, or storage internal to the laboratory.
- 6.8 Estimated Detection Limit (EDL): A calculation of the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2378-substituted congener that is not identified.
- 6.9 Estimated Maximum Possible Concentration (EMPC): A calculation for a peak characterized by a response with a signal-to-noise ratio of at least 2.5 for both the quantitation ions, and meeting all identification criteria except ion ratio. EMPC is a worst-case estimate of the concentration.
- 6.10 Extraction Standards: Isotopes added prior to extraction that serve as internal standards for many 2,3,7,8 substituted congeners. In addition, to measure the overall extraction and fractionation efficiencies. Method 8290A names them Internal Standards while Method 1613B uses the Labeled Compounds terminology.
- 6.11 Injection Standards: Isotopes added prior to injection to determine the recoveries of the Extraction and Cleanup Standards. Method 8290A names them Recovery Standards while Method 1613B calls them Internal Standards.
- 6.12 Internal Standard (ISTD): A known amount of standard added to a test portion of a sample as a reference for evaluating the retention time and concentration of dependent analytes and controlling the precision and bias of the applied analytical method.
- 6.13 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 6.14 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.
- 6.15 Matrix Spike and Matrix Spike Duplicate (MS and MSD): Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.
- 6.16 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

- 6.17 **Precision:** The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves, a data quality indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.
- 6.18 **Quantitation Limits (also PQL, RL):** The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the linear calibration range.
- 6.19 **Sampling Standards:** Isotopes added prior to field sampling for Method 0023A and Method TO-9a that are used to measure the efficiency of the sampling step alone.

7.0 INTERFERENCES/LIMITATIONS

- 7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.
- 7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.
- 7.3 Contamination may also occur in the GC/MS system. High boiling materials tend to build up in the injection port and the front end of the column. The analyst should maintain a thorough working knowledge of keeping the injection port free of contamination, including changing out the septum, injection port liner, O-ring, ferrule, and gold seal.
- 7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. If carryover is suspected, potentially impacted samples must be re-analyzed after any needed maintenance, solvent replacement, and/or cleaning has been done.

8.0 SAFETY PRECAUTIONS AND WARNINGS

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT. NO OCCUPATIONAL EXPOSURE LIMIT FOR DIOXIN HAS BEEN ESTABLISHED. IT IS A KNOWN AND PROBABLE HUMAN CARCINOGEN.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN USING METHYLENE CHLORIDE.

- 8.1 Eye protection should be worn when handling samples, reagents, or standards.
NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.
- 8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a current reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.
- 8.3 Personal Protective Equipment (PPE)
- 8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

- 8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.
- 8.5 Never leave gas cylinders unchained or untied.
- 8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.
- 8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.
- 8.8 The analyst must use care when assembling and operating instrumentation. Check to see that the gas chromatograph equipment is properly assembled and hooked up to the proper gas cylinder and power, referencing the appropriate manual. Analytical equipment must only be operated by qualified personnel.
- 8.9 For further safety instructions, consult the Safety Manual, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

- 9.1 Equipment associated with this method includes:
- 9.1.1 Gas tight syringes
- 9.1.2 2 mL high recovery (conical) autosampler vials and storage racks
- 9.1.3 Teflon crimp tops
- 9.1.4 Crimper/De-crimper
- 9.1.5 GC Columns
- 9.1.5.1 Agilent DB5-MS or equivalent; 60 m, 0.25 mm, 0.25 um
- 9.1.5.2 Agilent DB-225 or equivalent; 30 m, 0.25 mm, 0.25 um
- 9.1.6 Quartz/Glass injection port liners
- 9.1.7 Injection port liner O-ring seals
- 9.1.8 Gold seals
- 9.1.9 Ferrules
- 9.1.10 Column cleaving tool
- 9.1.11 Septa (thermogreen)
- 9.1.12 10-100 uL adjustable air displacement pipette with disposable tips
- 9.2 Instrumentation
- 9.2.1 Waters Autospec Premier high resolution mass spectrometer
- 9.2.2 Agilent 7890 Gas Chromatograph
- 9.2.2.1 A suggested temperature program for primary analysis follows:
- | | |
|---------------|------------|
| Initial Temp. | 140° C |
| Hold Time | 1.0 min. |
| Rate 1 | 20° C/min. |
| Temperature 2 | 180° C |

Time 2	2°/min
Temperature 3	235° C
Rate 3	30° C/min.
Final Temp.	290° C
Hold Time	13 min.
Run Time:	45 minutes (may vary due to column length or flow rate)
Solvent Delay:	18.0 min.
Splitless Valve Time:	1.5 min.
Flow:	1.8 mL/min.
Mass Range:	See descriptor definitions (Table 2)

NOTE: These instrument conditions and rates are guidelines which may change.

9.2.3 LEAP Technologies GC PAL Autosampler

9.2.3.1 Suggested parameters:

Sample volume – 1 µL

Air volume – 0.5 µL

Solvent push volume – 1 µL

Number of sample washes - 0

Solvent washes - 30

Sample viscosity wait – 1 second

Number of sample pumps - 0

Injection mode - Fast

10.0 REAGENTS AND STANDARDS

10.1 Reagents and standards

10.1.1 Nonane

10.1.2 Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These may include extraction standard, matrix spiking standard, cleanup standard, injection standard, as well as others. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference CF-LB-E-007 and CF-OA-E-002 for further information regarding standards and their preparation.

10.1.3 Initial Calibration (ICAL) Standards: Certified calibration standards are purchased from commercial vendors at a minimum of five concentration levels. One of the calibration standards is at a concentration near, but above, the method detection limit; the others should correspond to the expected range of compounds found in samples. Calibration standards expire after a maximum of five years and should be monitored frequently for signs of degradation.

10.1.4 Calibration Verification Standards (CVS, CCAL, CS3WT): A certified CVS is purchased from a second source commercial vendor at a concentration that is near to the midpoint of the calibration curve.

10.1.5 Window Defining Mix and Column Performance Mix (WDM and CPM): A standard containing the first and last eluters for each homolog group, as well as the dioxin and furan isomers used to demonstrate isomer specificity on the GC column in use. These may be contained in the same standard as the calibration verification (known as CS3WT).

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 Sample extracts have a 45-day holding time from the date of extraction by methods 8290A and 0023A, and a 365 day holding time from the date of extraction by 1613B. Note that per method 8290A, tissue extracts must be completely analyzed by 45 days from collection. TO-9a cartridges are considered clean for 30 days from preparation, samples must be extracted 7 days from collection and analyzed 40 days from extraction.

11.2 Sample extracts are delivered from the prep lab to the instrument lab and are stored in a darkened hood at room temperature. The extracts are usually grouped according to preparation batches and are accompanied by the batch pull sheet and other pertinent paperwork.

11.3 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to run into their custody prior to analysis.

11.4 All sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

12.1 Before extracts can be analyzed on the instrument, they must first be evaporated to dryness under nitrogen and then spiked with injection standard to set the final volume nominally at 20 μ L. A determination must also be made as to whether the extract should be diluted. The decision to dilute a sample extract is based on a number of factors: sample screening, historical data about the sample or sample site, the appearance of the extract (color, viscosity, incidental odor, turbidity, etc.), or regulatory considerations. The experience of the analyst is invaluable in making this determination.

NOTE: Sample extracts may contain multiple layers or sediment. Samples that contain sediment are returned to cleanup. Multiple layers are treated on a case-by-case basis. If the extract can be homogenized, then a uniform sample is achieved. If the extract remains bi-phasic, the PM and client are contacted for further guidance.

12.2 If a sample is to be analyzed without dilution ('neat'), 2 nanograms of injection standard solution is added to the extract using a pipette (20 μ L of a 0.1 ng/ μ L = 100 pg/ μ L extract concentration). A cap is then placed on the vial and secured by crimping before vortexing the sample to ensure complete mixing and vial wall washing.

12.3 If samples require dilution, the dilution is made using nonane or appropriate solvent. If not previously added, 2 nanograms of JS is added to the autosampler vial. Dilution prep may involve the addition of supplemental extraction standard (ES) and is documented in the injection prep logbook.

- 12.4 Once samples are prepped, they are ready to be injected onto the instrument. An autosampler is used to inject standards and sample extracts on the instrument.
- 12.5 The need for dilution may also be determined after analysis is performed, and may still be performed as above. Under normal circumstances, a sample would be diluted if any chromatographic peaks saturate the detector.

13.0 QUALITY CONTROL REQUIREMENTS

Typically a blank (LMB), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) are extracted and analyzed with each prep batch. Other client requirements may include a matrix spike (MS) and matrix spike duplicate (MSD) or sample duplicate (DUP).

13.1 Blanks

- 13.1.1 A blank is extracted with each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents and the analytical system are under control. Blanks are carried through all stages of sample preparation and analysis. For Method 1613B, an acceptable blank must be below the minimum levels listed in Table 2 of the method for all analytes. For Methods 8290A, 0023A, and TO-9a, all analytes must be below the Lower Method Calibration Limits.
- 13.1.2 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 6 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for hepta- and OCDD. Sampling standards for Method TO-9a must be within 70-130%.

13.2 Laboratory Control Samples and Matrix Spikes

- 13.2.1 The spiking standard for LCS/LCSDs and MS/MSDs contains all analytes listed in Table 4. For each LCS, LCSD, MS and MSD, the concentration of each analyte and its percent recovery are calculated as shown in Sec. 17.4.1 and 17.4.5. For methods 8290A and 0023A, percent recoveries should be within 70-130%. For method 1613B, recovered concentrations should be within the limits in Table 5.
- 13.2.2 If recovery is not within these limits, the data may need to be re-checked for errors, or the samples and QC may need to be re-analyzed. In addition, the instrumentation may need to be checked for performance problems. If the LCS fails to meet acceptance criteria due to low recovery, the associated samples may have to be re-extracted and re-analyzed when possible. If one or more recoveries are high in the LCS and these analytes are not detected in the samples, the event should be documented and data may be reported. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.

NOTE: Many clients have contract specific criteria that must be considered when evaluating recovery of the Quality Control samples.

13.2.3 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 5 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for hepta- and OCDD. Sampling standards for Method TO-9a must be within 70-130%.

13.3 Samples

13.3.1 The percent recovery of each labeled standard (as listed in SOP CF-OA-E-001) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 5 for method 1613B or 40-135% for method 8290A. For method TO-9a, extraction standards must be within 50-120% for tetra- through hexa- and within 40-120% for hepta- and OCDD. Sampling standards for Method TO-9a must be within 70-130%.

13.3.2 Calculated EDLs should be below the PQLs in Table 1. Any reported EDLs above the PQLs should be noted in the case narrative.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

14.1 Mass spectrometer performance

14.1.1 The mass spectrometer is operated in electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. Corrective action must be implemented whenever the resolving power does not meet the requirement.

14.1.1.1 Chromatography time for PCDDs and PCDFs exceeds the long term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass drift correction is mandatory. A lock-mass ion from the reference compound PFK is used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Lock mass ions may be found in the descriptor table, Table 2. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full scale deflection for a given set of detector parameters. Under these conditions, sensitivity changes that might occur during the analysis can be more effectively monitored. NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

14.1.2 Documentation of the instrument resolving power must be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 2) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10 percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

14.2 System Performance

System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 9.1. The laboratory must document that all applicable system performance criteria are met before sample analysis begins. Sec. 9.2.2 provides recommended GC conditions that may be used to satisfy the required criteria. Mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is required at the beginning of each 12-hr period during which samples are analyzed. For Method 1613B, a continuing calibration must be performed at the beginning of the sequence, while for Methods 0023A and 8290A, continuing calibrations must be performed at both the beginning and the end of a sequence. An ending continuing calibration may also serve as the beginning check for the next sequence.

14.2.1 GC Column performance check

14.2.1.1 Inject 1 μ L of an aliquot of the column performance check solution (Sec. 10.1.5) and acquire selected ion monitoring (SIM) data within a total cycle time of ≤ 1 second. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of ≤ 25 percent (Figure 1), where:

$$\text{Valley percent} = (x/y) \times 100$$

x = measured as in Figure 1 from the 2,3,7,8-closest TCDD eluting isomer

y = the peak height of 2,3,7,8-TCDD

For 2378-TCDF confirmatory analysis, the chromatographic separation between 2378-TCDF and its closest eluters must be resolved with a valley of ≤ 25 percent.

14.2.1.2 It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions described in this SOP. Their retention times are used

to determine the five homologue retention time windows that are used for qualitative (Sec. 15.3.1.1) and quantitative purposes. All peaks (including $^{13}\text{C}_{12}$ -2,3,7,8-TCDD) should be labeled and identified on the chromatograms. All first eluters of a homologous series should be labeled with the letter "F," and all last eluters of a homologous series should be labeled with the letter "L". Any individual selected ion current profile (SICP) or the reconstructed homologue ion current constitutes an acceptable form of data presentation. A SICP for the labeled compounds is also required.

14.2.1.3 Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (1,2,8,9-TCDF) and the first penta-chlorinated congener (1,3,4,6,8-PeCDF), as these two compounds elute within 15 sec of each other on the 60-m DB-5 column, and overlap on the 60-m DB-5ms column. Both congeners must be acquired within one analysis.

14.2.1.4 The absolute retention time of $^{13}\text{C}_{12}$ -1,2,3,4-TCDD must exceed 25.0 minutes on the primary GC column in use, and 15.0 minutes on the confirmatory GC column.

14.3 Initial Calibration

14.3.1 Prior to running a multi-level calibration, take precautions to ensure that the instrument meets system performance criteria. The analyst must document that all system performance criteria are met before analyzing an initial calibration.

14.3.2 Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria listed below. Initial calibration is also required if any routine calibration does not meet the required criteria listed in Sec. 15.2, and at a minimum, annually.

14.3.3 At a minimum, all five high-resolution concentration calibration solutions listed in Table 4 must be used for the initial calibration.

14.3.4 Tune the instrument with PFK to meet the above-specified system performance criteria.

14.3.5 Inject the GC column performance check solution and acquire SIM mass spectral data. The total cycle time for each descriptor must be < 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 15.1.1.1 are met.

14.3.6 By using the same conditions (GC and MS) that produced acceptable results with the column performance check solution, analyze each of the five concentration calibration solutions. Each injection must meet the following ion ratio and signal-to-noise (S/N) requirements:

14.3.6.1 The ratio of the areas of the integrated ion current for the ions appearing in Table 2 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) in Table 3. These ion ratio requirements must be within the specified control limits simultaneously in one run. It is the analyst's

responsibility to take corrective action if the ion abundance ratios are outside the limits.

- 14.3.6.2 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the S/N ratio must be better than or equal to 10. Manual measurement of S/N is required for any GC peak that has an apparent S/N of less than 15:1. The result of the calculation must appear on the SICP above the GC peak in question.
- 14.3.7 Calculate the 17 relative response factors (RF) for unlabeled target analytes relative to their appropriate internal standards (see Table 10). Also calculate the RFs for the ESs and CSs relative to the appropriate injection standards according to the following formula:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Sum of the Areas of the two characteristic ions for the compound being measured.

A_{is} = Sum of the Areas of the two characteristic ions for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

The RF is a dimensionless quantity; the units used to express C_{is} and C_x must be the same.

- 14.3.8 The RF for other isomers within a homolog group shall be determined from the average RF of the 2,3,7,8-substituted isomers. For example, the RF for non-2,3,7,8-substituted HxCDD isomers (totals peaks) is the average of the three 2,3,7,8-substituted isomers. NOTE: If only one 2,3,7,8-substituted isomer is present in the calibration then use that isomer's RF for all isomers within its homolog group.
- 14.3.9 Because more than five calibration levels may be analyzed, the analyst may choose to deactivate one or more levels globally. If a level is not used, it will be deactivated in the method for all analytes in that calibration mixture. In some cases the upper level(s) of the calibration may be deactivated in order to meet method criteria for single compounds. This practice results in a narrower calibration range. The low standard representing the PQL cannot be dropped. Please note that this practice does not represent "cherry picking," which is acknowledged as an unacceptable laboratory practice.
- 14.3.10 The average RF must be calculated for each compound as follows:

$$RF_{avg} = \frac{\sum_{i=1}^n X}{n}$$

Where:

N = number of calibration levels

X_i ; $i=1$ to n , are the compounds RF values for each calibration point

14.3.11 Criteria for acceptable initial calibration

The criteria listed below for acceptable calibration must be met before sample analyses are performed.

14.3.11.1 Per method 8290A, the percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the nine labeled reference compounds must not exceed ± 20 percent. These limits also apply to Method 0023A. Per method 1613B, the percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the fifteen labeled reference compounds must not exceed ± 35 percent. See Table 12 for method TO-9a minimum requirements.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = relative standard deviation

\bar{x} = mean of 5 or more initial RFs for a compound

SD = standard deviation of average RFs for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^n (X - A)^2}{n - 1}}$$

where:

n = number of calibration levels

X_i ; $i=1$ to n , are the compounds RF values for each calibration point

A = average of the RFs from above

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

15.1 Resolution check

15.1.1 At the beginning and end of each 12-hour window, mass resolution must be tuned and/or verified. A static resolving power of at least 10,000 must be demonstrated at appropriate masses before analysis is performed.

15.1.2 Using a PFK molecular leak, tune the instrument to the minimum required resolving power of 10,000 at m/z 330.9792 (for day to day operations, the instrument may be tuned to approximately 11,000). Verify that the exact mass of m/z 380.9760 is within 5 ppm of the required value.

15.2 Column Performance/Window Defining/Continuing Calibration Check (CS3WT)

15.2.1 Inject 1 uL of the CS3WT or CPM. Verify that all column performance and window defining criteria in Section 14.2.1 have been met.

15.2.2 The CS3WT also contains the analytes for continuing calibration. The initial calibration curve for each compound of interest must be verified once every 12 hours.

Calculate the percent difference using:

$$\% \text{ Difference} = \frac{|\overline{RF}_i - RF_c|}{\overline{RF}_i} \times 100$$

Where:

\overline{RF}_i = average response factor from initial calibration

RF_c = response factor from current CS3WT

Calculate analyte concentrations using:

$$[PCDD / PCDF] = \frac{(A_{unk}^{ion1} + A_{unk}^{ion2})}{(A_{ES}^{ion1} + A_{ES}^{ion2})} \times \frac{Q_{ES}}{RF}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.

Q_{ES} = the amount of extraction standard in pg/uL

RF = Average RF from the ICAL for the compound

15.2.2.1 For methods 0023A and 8290A, if the percent difference for each native analyte in the CS3WT is $\leq 20\%$, and for each labeled analyte is $\leq 30\%$, the initial calibration is assumed to be valid. For method 1613B, analyte concentrations must fall within the limits in Table 7. If the criteria are not met, corrective action should be taken. If no source of the problem can be determined after corrective action has been taken, a new calibration may need to be generated. For Method TO-9a See Table 12 for minimum requirements.

15.2.2.2 All ion ratios must be within the limits in Table 3.

15.2.2.3 For methods 0023A and 8290A, if no more than two unrelated compounds in the continuing calibration check performed at the end of a 12-hour period fail by no more than $\pm 25\%$ for the 17 unlabeled compounds and $\pm 35\%$ for the 9 labeled compounds, the average RF values from the beginning and ending continuing calibration checks should be used to compute the analyte concentrations, instead of the RF values obtained from the initial calibration. No further sample analyses should be performed until an acceptable calibration is achieved.

15.3 Sample Analysis

15.3.1 Data Interpretation

15.3.1.1 Qualitative Determination

For a peak to be identified as a PCDD or PCDF, it must meet all of the criteria listed below.

- 15.3.1.1.1 The signals for the two m/z's being monitored must be present and maximize within ± 2 seconds of each other.
 - 15.3.1.1.2 The signal-to-noise ratio between the two m/z's must be ≥ 2.5 for native compounds and ≥ 10 for labeled compounds.
 - 15.3.1.1.3 Ion ratios must be within the limits in Table 3.
 - 15.3.1.1.4 Relative Retention Times
 - 15.3.1.1.4.1 For Methods 0023A and 8290A, congeners which have an isotopically labeled compound must fall within -1 to +3 seconds of the labeled compound. Congeners with no labeled compound must be within 0.005 retention time units of the RRT measured in the continuing calibration. (See Table 11.) For method TO-9a, congeners which have an isotopically labeled compound must fall within -3 to +3 seconds of the labeled compound. Congeners with no labeled compound must be within 0.005 retention time units of the RRT measured in the continuing calibration.
 - 15.3.1.1.4.2 For Method 1613B, relative retention times must be within the RRT limits found in Table 8.
 - 15.3.1.1.4.3 For non-2378 peaks, retention times must be within the retention time windows established by the analysis of the window defining mixture (Sec. 14.2.1.2).
 - 15.3.1.1.5 For PCDFs, no peak may be present in the associated PCDPE channel at the same retention time. If a PCDPE peak is present, the PCDF peak should be reported with a flag denoting the interference.
 - 15.3.1.1.6 Any sample in which 2378-TCDF has been identified at or above the method reporting limit must be confirmed on a second column (DB-225 or equivalent).
- 15.3.1.2 Calibration Limit Exceedance
- 15.3.1.2.1 If a compound in a sample exceeds the upper calibration limit, all subsequent samples must be checked for carryover contamination.

15.3.1.2.2 When a subsequent sample is non-detect for the compound in question, the sequence is again considered acceptable for reporting.

15.3.1.2.3 All affected samples between the exceeding sample and the non-detect sample must be re-analyzed.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Preventive maintenance on a HRGC/HRMS system involves the following basic areas:

16.1.1 Vacuum pumps for the inlets, source, and analyzer need a change of oil about every year or when system performance indicates it is needed.

16.1.2 The GC injection port is cleaned as needed, approximately once a week. It is recommended that the septum and injection port liner be replaced at the time of cleaning. Additionally, the gold plated seal should be cleaned or replaced.

16.1.3 Ion source maintenance is usage dependent. The type and quantity of samples that have been injected determine the frequency of ion source cleaning and filament replacement.

16.1.4 Autosampler maintenance is primarily that of cleanliness. Most autosamplers need their moving parts to be clean and lightly lubricated. The most frequent corrective maintenance is that of changing the syringe, usually about once per month.

16.1.5 Instrument maintenance logs are kept with each instrument and serve as a record of all the maintenance that has been done on the instrument.

16.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

16.2.1 Service is provided to the instrument via the analyst, the in-house instrument service engineer, or a technical support specialist from the manufacturer. When instrument failure occurs, different parts of the instrument are isolated to determine the root cause. For example, the injection port may be capped off if a leak is suspected to prove the leak is/is not coming from that source. Instrument maintenance logbooks are kept for each instrument detailing the type of maintenance performed on the instrument and when it was performed. Preventive maintenance visits are scheduled annually for the mass spectrometers.

16.2.2 Analytical GC columns are clipped or replaced when the existing column shows signs of excessive degradation or the inability to properly resolve chromatographic peaks. Excessive peak tailing, poor responses, and baseline disturbances may also indicate that the column needs to be replaced.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data are evaluated qualitatively and quantitatively using a software program such as Waters MassLynx, or equivalent data system.

17.2 Data are reviewed, and a hard copy is generated. If manual integrations are made, a hard copy of the manual integration is printed and initialed by the analyst and included with the raw data.

17.3 Additional supporting documentation, such as totals pages generated by the software may be included with the data.

17.4 Quantitative Analysis

17.4.1 The concentration (ng/L for aqueous, ng/g for solids) of each identified compound in the sample is calculated as follows:

$$[PCDD / PCDF] = \frac{(A_{unk}^{ion1} + A_{unk}^{ion2})}{(A_{ES}^{ion1} + A_{ES}^{ion2})} \times \frac{Q_{ES}}{W_{unk} \times D \times \overline{RF}}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.

Q_{ES} = the amount of extraction standard added to the sample in nanograms

W_{unk} = the initial sample aliquot size, in liters for waters and in grams for solids.

D = (% moisture in sample)/100, or 1 for waters

\overline{RF} = Average RF from the ICAL for the compound

17.4.2 The estimated detection limit (EDL) is calculated as follows:

$$[EDL_{ppt}] = 3 \times \frac{(H_{unk}^{ion1} + H_{unk}^{ion2})}{(H_{ES}^{ion1} + H_{ES}^{ion2})} \times \frac{Q_{ES}}{W_{unk} \times \overline{RF}}$$

Where:

H_{unk} = the height of the noise present in each ion monitored.

H_{ES} = the height of the extraction standard peak in each ion monitored.

3 = signal-to-noise factor for minimum height of peak.

17.4.3 The estimated maximum possible concentration (EMPC) is calculated in the same manner as a concentration (Section 17.4.1).

17.4.4 The concentration of each extraction and cleanup standard is calculated as follows:

$$[ES_{ng}] = \frac{(A_{ES}^{ion1} + A_{ES}^{ion2})}{(A_{JS}^{ion1} + A_{JS}^{ion2})} \times \frac{Q_{JS}}{\overline{RF}}$$

Where:

A_{ES} and A_{JS} = the integrated area for each ion monitored.

Q_{JS} = the amount of injection standard added to the sample in nanograms

\overline{RF} = Average RF from the ICAL for the compound

The cleanup standard concentration is calculated as above, substituting the area of the individual cleanup standard ions for the extraction standard ions.

17.4.5 Percent recovery is calculated as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

Where:

R_{ng} = the amount of standard recovered in nanograms.

S_{ng} = the amount of standard spiked in nanograms.

18.0 POLLUTION/CONTAMINATION

- 18.1 Work area should be maintained free of dust and dirt accumulations.
- 18.2 Fume hoods are utilized to remove fumes and reduce the risk of airborne contaminants to ensure personnel safety. Hoods are monitored in accordance with CF-FC-E-003 for Fume Hood Face Velocity Performance Checks.
- 18.3 The laboratory area is restricted to authorized personnel.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

- 19.1 A review process is used to insure the quality of the data. Raw data are reviewed first by the analyst, then by a second (peer) analyst or a data validator. When the analyst is satisfied that the data have been correctly processed and uploaded to the LIMS, a data report is generated from AlphaLIMS. The AlphaLIMS report along with the raw data and supporting documentation, such as a run log and case narrative, are submitted for review to the data validator or another experienced analyst. The reviewer goes through the raw data as if he/she was working it up for the first time and verifies that they are correct. In addition, he/she must make sure that the data have been correctly entered into AlphaLIMS. AlphaLIMS reports may be self-reviewed. If errors are discovered in either the raw data or the AlphaLIMS report, then the two analysts should discuss the differences and how best to resolve them. In some cases, the peer review process may uncover errors that lead to a sample being re-extracted or re-run. In cases such as these, a nonconformance report (NCR) should be completed and submitted to the Quality department. It is recommended that a copy of the NCR be given to the prep analyst if it involves a re-extraction and that a copy be kept with the original data.
- 19.2 Once the data review has been completed by the reviewer, the batch is returned to the analyst for corrections (if applicable) and the status is updated from REVW to DONE in AlphaLIMS.
- 19.3 Data may be transmitted automatically to AlphaLIMS. This automatic "upload" procedure may be activated prior to data review or after data review is complete. In either case, the data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-analysis, re-extraction, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a nonconformance report and/or corrective action should be initiated in accordance with CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when an initial or continuing calibration, blank and/or laboratory control sample fails to meet the acceptance criteria defined for the method. Troubleshooting may involve one or more of the following actions:

- 21.1 If analytes in a multi-point calibration fail to meet specified criteria, additional standards for the failing compounds may need to be reanalyzed. If they still do not

meet specifications, instrument maintenance or new standards may be required before work is continued.

- 21.2 If a continuing calibration fails to meet specified criteria, instrument tuning or inlet maintenance may be required. If routine maintenance procedures fail to produce a second consecutive calibration verification within acceptance criteria, then the laboratory must demonstrate acceptable performance after further corrective action with two consecutive calibration verifications, or a new initial calibration must be analyzed.
- 21.3 If a method blank fails to meet defined criteria, the source of contamination should be found and eliminated before proceeding with analysis.
- 21.4 If normal equipment and software operating procedures do not resolve troubleshooting efforts, the manuals for software, hardware and other equipment discussed in this SOP are available for consultation and resolution. On-line support may be available from software and instrument manufacturers, as well. Any revisions, repairs or corrective actions required must be documented in accordance with the laboratory's Quality System as described in CF-QS-B-001.

22.0 RECORDS MANAGEMENT

- 22.1 Run logs are generated for each instrument each day that the instrument is run. These run logs serve as records of what is run on the instrument, including samples, QC, calibrations, tunes, etc. Additional information is provided in the run log, including the analyst's initials, run date and time, and file name.
- 22.2 Raw data are stored in the lab in filing cabinets and/or boxes as long as there is space available. When space runs out, the data are boxed and sent to storage.
- 22.3 All records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Sample extracts that have been run are temporarily stored in case they have to be reanalyzed. Once space is no longer available to keep them in the lab, they are moved to Waste Disposal where they are handled and disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

24.0 REFERENCES

- 24.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 8290A, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry (HRGC/HRMS)," Rev. 1, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.
- 24.2 Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Rev. B, Oct. 1994. USEPA, Office of Water, Engineering and Analysis Division, 401 M Street SW, Washington, D.C. 20460.
- 24.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 0023A, "Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran

Emissions From Stationary Sources,” Rev. 1, Dec. 1996. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

- 24.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition. “Compendium Method TO-9A, Determination of Polychlorinated, Polybrominated and Brominated/Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air.” January 1999. Center for Environmental Research Information, Office of Research and Development, USEPA, Cincinnati, OH 45268.
- 24.5 National Environmental Laboratory Accreditation Conference (NELAC) Standard, July 2003.

25.0 HISTORY

Revision 1: Section 15.3.1.2 added.

Revision 2: Absolute RT information added in 14.2.1.4; Calibration limit exceedance information added in section 15.3.1.2; Table 8 footnote describing RRT window adjustment to column used.

Revision 3: Method 0023A requirements added.

Revision 4: 2378-TCDF confirmation procedure and requirements added.

Revision 5: Injection standard changed from Tridecane to nonane. Discussion of equipment use and operation instructions was added, per DoD ELAP gray box 22.

Revision 6: Added TO-9a support and additional Tables for Method 8290.

Revision 7: Removed references to 8290 cleanup standard. Added TO-9a reference.

TABLE 1: METHOD ANALYTES AND PQLs

Analyte	Solid/Tissues (pg/g)	Aqueous (pg/L)	Air (pg)	CAS Number*
2378-TCDD	1	10	10	1746-01-6
12378-PeCDD	5	50	50	40321-76-4
123478-HxCDD	5	50	50	39227-28-6
123678-HxCDD	5	50	50	57653-85-7
123789-HxCDD	5	50	50	19408-74-3
1234678-HpCDD	5	50	50	35822-39-4
OCDD	10	100	100	3268-87-9
2378-TCDF	1	10	10	51207-31-9
12378-PeCDF	5	50	50	57117-41-6
23478-PeCDF	5	50	50	57117-31-4
123478-HxCDF	5	50	50	70648-26-9
123678-HxCDF	5	50	50	57117-44-9
234678-HxCDF	5	50	50	60851-34-5
123789-HxCDF	5	50	50	72918-21-9
1234678-HpCDF	5	50	50	67562-39-4
1234789-HpCDF	5	50	50	55673-89-7
OCDF	10	100	100	39001-02-0

* Chemical Abstract Services number

TABLE 2: MASS DESCRIPTORS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	50	10
1	2	305.8987	50	10
1	3	315.9419	50	10
1	4	304.9824	50	10
1	5	304.9824	(Lock)	10
1	6	317.9389	50	10
1	7	319.8965	50	10
1	8	321.8936	50	10
1	9	327.8847	50	10
1	10	331.9368	50	10
1	11	333.9339	50	10
1	12	339.8597	50	10
1	13	341.8568	50	10
1	14	375.8364	50	10
2	1	339.8597	50	10
2	2	341.8568	50	10
2	3	351.9	50	10
2	4	353.897	50	10
2	5	355.8546	50	10
2	6	357.8517	50	10
2	7	366.9792	50	10
2	8	366.9792	(Lock)	10
2	9	367.8949	50	10
2	10	369.8919	50	10
2	11	409.7974	50	10
3	1	373.8207	50	10
3	2	375.8178	50	10
3	3	380.976	50	10

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
3	4	380.976	(Lock)	10
3	5	383.8639	50	10
3	6	385.861	50	10
3	7	389.8156	50	10
3	8	391.8127	50	10
3	9	401.8559	50	10
3	10	403.853	50	10
3	11	445.7555	50	10
4	1	407.7818	50	10
4	2	409.7788	50	10
4	3	417.8253	50	10
4	4	419.822	50	10
4	5	423.7767	50	10
4	6	425.7737	50	10
4	7	430.9728	50	10
4	8	430.9728	(Lock)	10
4	9	435.8169	50	10
4	10	437.814	50	10
4	11	479.7165	50	10
5	1	441.7427	50	10
5	2	443.7398	50	10
5	3	454.9728	50	10
5	4	454.9728	(Lock)	10
5	5	457.7377	50	10
5	6	459.7348	50	10
5	7	469.778	50	10
5	8	471.775	50	10
5	9	513.6775	50	10

TABLE 3: THEORETICAL ION RATIOS AND CONTROL LIMITS

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6 ^a	0.51	0.43	0.59
7	1.05	0.88	1.20
7 ^b	0.44	0.37	0.51
8	0.89	0.76	1.02

^a Used only for ¹³C-HxCDF

^b Used only for ¹³C-HpCDF

TABLE 4: 1613B LIMITS FOR TETRA ONLY TESTS

Compound Name	Test Conc. (pg/μL)	CCAL Limits (pg/μL)	OPR Limits (pg/μL)	Sample Limits (pg/μL)
2,3,7,8-TCDD	10	8.2 - 12.3	7.3 - 14.6	-
2,3,7,8-TCDF	10	8.6 - 11.6	8.0 - 14.7	-
¹³ C ₁₂ -2,3,7,8-TCDD	100	85 - 117	25 - 141	31 - 137
¹³ C ₁₂ -2,3,7,8-TCDF	100	76 - 131	26 - 126	29 - 140
³⁷ Cl ₄ -2,3,7,8-TCDD	10	8.3 - 12.1	3.7 - 15.8	4.2 - 16.4

TABLE 5: INITIAL CALIBRATION CONCENTRATIONS

Analyte	Concentration (pg/uL)				
	CS-0.5	CS-2	CS-3	CS-4	CS-5
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
<u>Extraction Standards</u>					
¹³ C-2378-TCDD	100	100	100	100	100
¹³ C-2378-TCDF	100	100	100	100	100
¹³ C-12378-PeCDD	100	100	100	100	100
¹³ C-12378-PeCDF	100	100	100	100	100
¹³ C-23478-PeCDF	100	100	100	100	100
¹³ C-123678-HxCDD	100	100	100	100	100
¹³ C-123478-HxCDD	100	100	100	100	100
¹³ C-123478-HxCDF	100	100	100	100	100
¹³ C-123678-HxCDF	100	100	100	100	100
¹³ C-123789-HxCDF	100	100	100	100	100
¹³ C-234678-HxCDF	100	100	100	100	100
¹³ C-1234678-HpCDD	100	100	100	100	100
¹³ C-1234678-HpCDF	100	100	100	100	100
¹³ C-1234789-HpCDF	100	100	100	100	100
¹³ C-OCDD	200	200	200	200	200
<u>Cleanup Standards</u>					
³⁷ Cl-2378-TCDD	0.25	2	10	40	200
<u>Injection Standards</u>					
¹³ C-1234-TCDD	100	100	100	100	100
¹³ C-123789-HxCDD	100	100	100	100	100

TABLE 6: METHOD 1613B LCS LIMITS

LCS Recovery Limits		
Analyte	Amount Spiked	Limit
	(pg/uL)	(pg/uL)
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
¹³ C-2378-TCDD	100	20-175
¹³ C-12378-PeCDD	100	21-227
¹³ C-123478-HxCDD	100	21-193
¹³ C-123678-HxCDD	100	25-163
¹³ C-1234678-HpCDD	100	26-166
¹³ C-OCDD	200	26-397
¹³ C-2378-TCDF	100	22-152
¹³ C-12378-PeCDF	100	21-192
¹³ C-23478-PeCDF	100	13-328
¹³ C-123478-HxCDF	100	19-202
¹³ C-123678-HxCDF	100	21-159
¹³ C-123789-HxCDF	100	17-205
¹³ C-234678-HxCDF	100	22-176
¹³ C-1234678-HpCDF	100	21-158
¹³ C-1234789-HpCDF	100	20-186
³⁷ Cl-2378-TCDD	10	3.1-19.1

TABLE 7: METHOD 1613B ES (SAMPLES & LMB) RECOVERY LIMITS

Compound Name	Amount Spiked (pg/ μ L)	Limits %
¹³ C ₁₂ -2,3,7,8-TCDD	100	25 - 164
¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	25 - 181
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	100	32 - 141
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	28 - 130
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	23 - 140
¹³ C ₁₂ -OCDD	200	17 - 157
¹³ C ₁₂ -2,3,7,8-TCDF	100	24 - 169
¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	24 - 185
¹³ C ₁₂ -2,3,4,7,8-PeCDF	100	21 - 178
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	100	26 - 152
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	26 - 123
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	100	28 - 136
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	100	29 - 147
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	28 - 143
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	100	26 - 138
³⁷ Cl ₄ -2,3,7,8-TCDD	10	35 - 197

TABLE 8: METHOD 1613B CONTINUING CALIBRATION LIMITS

Compound Name	CCAL (pg/ μ L)	Limits (pg/ μ L)	Compound Name	CCAL (pg/ μ L)	Limits (pg/ μ L)
2,3,7,8-TCDD	10	7.8 - 12.9	¹³ C ₁₂ -2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	¹³ C ₁₂ -1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	¹³ C ₁₂ -1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	¹³ C ₁₂ -1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	¹³ C ₁₂ -OCDD	200	96 - 415
OCDD	100	79 - 126	¹³ C ₁₂ -2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	¹³ C ₁₂ -1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	¹³ C ₁₂ -2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	¹³ C ₁₂ -1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	¹³ C ₁₂ -1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	¹³ C ₁₂ -2,3,4,6,7,8-HxCDF	100	73 - 137
2,3,4,6,7,8-HxCDF	50	44 - 57	¹³ C ₁₂ -1,2,3,7,8,9-HxCDF	100	74 - 135
1,2,3,7,8,9-HxCDF	50	45 - 56	¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	³⁷ Cl ₄ -2,3,7,8-TCDD	10	7.9 - 12.7
OCDF	100	63 - 159			

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TABLE 9: METHOD 1613B RELATIVE RETENTION TIME LIMITS

Compound	RRT Reference	RRT Limits
2,3,7,8-TCDF	13C -2,3,7,8-TCDF	0.999-1.003
2,3,7,8-TCDD	13C -2,3,7,8-TCDD	0.999-1.002
1,2,3,7,8-PeCDF	13C -1,2,3,7,8-PeCDF	0.999-1.002
2,3,4,7,8-PeCDF	13C -2,3,4,7,8-PeCDF	0.999-1.002
1,2,3,7,8-PeCDD	13C -1,2,3,7,8-PeCDD	0.999-1.002
1,2,3,4,7,8-HxCDF	13C -1,2,3,4,7,8-HxCDF	0.999-1.001
1,2,3,6,7,8-HxCDF	13C -1,2,3,6,7,8-HxCDF	0.997-1.005
1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDF	0.999-1.001
2,3,4,6,7,8-HxCDF	13C -2,3,4,6,7,8,-HxCDF	0.999-1.001
1,2,3,4,7,8-HxCDD	13C -1,2,3,4,7,8-HxCDD	0.999-1.001
1,2,3,6,7,8-HxCDD	13C -1,2,3,6,7,8,-HxCDD	0.998-1.004
1,2,3,7,8,9-HxCDD	13C -1,2,3,6,7,8,-HxCDD	1.000-1.019
1,2,3,4,6,7,8-HpCDF	13C -1,2,3,4,6,7,8-HpCDF	0.999-1.001
1,2,3,4,7,8,9-HpCDF	13C -1,2,3,4,7,8,9-HpCDF	0.999-1.001
1,2,3,4,6,7,8-HpCDD	13C -1,2,3,4,6,7,8-HpCDD	0.999-1.001
OCDF*	13C -OCDD	1.004-1.013
OCDD	13C -OCDD	0.999-1.001
13C -2,3,7,8-TCDF	13C -1,2,3,4-TCDD	0.923-1.103
13C -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	0.976-1.043
37Cl -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	0.989-1.052
13C -1,2,3,7,8-PeCDF	13C -1,2,3,4-TCDD	1.000-1.425
13C -2,3,4,7,8-PeCDF	13C -1,2,3,4-TCDD	1.011-1.526
13C -1,2,3,7,8-PeCDD	13C -1,2,3,4-TCDD	1.000-1.567
13C -1,2,3,4,7,8-HxCDF*	13C -1,2,3,7,8,9-HxCDD	0.958-0.984
13C -1,2,3,6,7,8-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.949-0.975
13C -1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.977-1.047
13C -2,3,4,6,7,8,-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.959-1.021
13C -1,2,3,4,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.977-1.000
13C -1,2,3,6,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.981-1.003
13C -1,2,3,4,6,7,8-HpCDF	13C -1,2,3,7,8,9-HxCDD	1.043-1.085
13C -1,2,3,4,7,8,9-HpCDF	13C -1,2,3,7,8,9-HxCDD	1.057-1.151
13C -1,2,3,4,6,7,8-HpCDD*	13C -1,2,3,7,8,9-HxCDD	1.074-1.098
13C -OCDD	13C -1,2,3,7,8,9-HxCDD	1.032-1.311

*** Due to the use of the DB-5MS column, these compounds exhibit slightly different elution times, resulting in RRT limits which vary from the method.**

TABLE 10: Method 8290 IS assignments

**Internal Standard References
Method 8290**

Analytes	Internal Standards
2378-TCDD	¹³ C-2378-TCDD
12378-PeCDD	¹³ C-12378-PeCDD
123478-HxCDD	¹³ C-123678-HxCDD
123678-HxCDD	¹³ C-123678-HxCDD
123789-HxCDD	¹³ C-123678-HxCDD
1234678-HpCDD	¹³ C-1234678-HpCDD
OCDD	¹³ C-OCDD
2378-TCDF	¹³ C-2378-TCDF
12378-PeCDF	¹³ C-12378-PeCDF
23478-PeCDF	¹³ C-12378-PeCDF
123478-HxCDF	¹³ C-123678-HxCDF
123678-HxCDF	¹³ C-123678-HxCDF
123789-HxCDF	¹³ C-123678-HxCDF
234678-HxCDF	¹³ C-123678-HxCDF
1234678-HpCDF	¹³ C-1234678-HpCDF
1234789-HpCDF	¹³ C-1234678-HpCDF
OCDF	¹³ C-OCDD
Extraction Standards	Injection Standards
¹³ C-2378-TCDD	¹³ C-1234-TCDD
¹³ C-12378-PeCDD	¹³ C-1234-TCDD
¹³ C-123678-HxCDD	¹³ C-123789-HxCDD
¹³ C-1234678-HpCDD	¹³ C-123789-HxCDD
¹³ C-OCDD	¹³ C-123789-HxCDD
¹³ C-2378-TCDF	¹³ C-1234-TCDD
¹³ C-12378-PeCDF	¹³ C-1234-TCDD
¹³ C-123678-HxCDF	¹³ C-123789-HxCDD
¹³ C-1234678-HpCDF	¹³ C-123789-HxCDD
Injection Standards	
¹³ C-1234-TCDD	NA
¹³ C-123789-HxCDD	NA

TABLE 11: 8290 Retention time limits**Retention Time Limits****Method 8290**

Analytes	Description	Limits
2378-TCDD	2,3,7,8-substituted congeners, which have an isotopically-labeled standard present in the sample extract	must be within -1 to +3 seconds of the isotopically-labeled standard
12378-PeCDD		
123678-HxCDD		
123789-HxCDD		
1234678-HpCDD		
OCDD		
2378-TCDF		
12378-PeCDF		
123678-HxCDF		
123789-HxCDF		
1234678-HpCDF		
123478-HxCDD	2,3,7,8-substituted compounds that do not have an isotopically-labeled standard present in the sample extract	must fall within 0.005 retention time units of the relative retention time as determined from the daily routine calibration
23478-PeCDF		
123478-HxCDF		
234678-HxCDF		
1234789-HpCDF		
OCDF	Non-2,3,7,8-substituted target compounds	must be within the corresponding homologous retention time windows established by analyzing the column performance check solution, relative to an isotopically-labeled standard in the sample
Total TCDDs		
Total PeCDDs		
Total HxCDDs		
Total HpCDDs		
Total TCDFs		
Total PeCDFs		
Total HxCDFs		
Total HpCDFs	Isotopically-labeled standards	No method limits: allowed to shift as long as the predicted RT of the native window defining isomers established by analyzing the column performance check solution remain within the descriptor switching time
¹³ C-2378-TCDD		
¹³ C-12378-PeCDD		
¹³ C-123678-HxCDD		
¹³ C-1234678-HpCDD		
¹³ C-OCDD		
¹³ C-2378-TCDF		
¹³ C-12378-PeCDF		
¹³ C-123678-HxCDF		
¹³ C-1234678-HpCDF		
¹³ C-1234-TCDD		

¹³C-123789-HxCDD**TABLE 12: METHOD TO-9A MINIMUM REQUIREMENTS FOR INITIAL AND DAILY****CALIBRATION**

Unlabeled Analytes	ICAL (RSD)	CVS (%D)
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25
OCDF	30	30

Internal Standards

13C-2,3,7,8-TCDD	25	25
13C-1,2,3,7,8-PeCDD	30	30
13C-1,2,3,6,7,8-HxCDD	25	25
13C-1,2,3,4,6,7,8- HpCDD	30	30
13C-OCDD	30	30
13C-2,3,7,8-TCDF	30	30
13C-1,2,3,7,8-PeCDF	30	30
13C-1,2,3,4,7,8-HxCDF	30	30
13C-1,2,3,4,6,7,8- HpCDF	30	30

Surrogate Standards

37Cl-2,3,7,8-TCDD	25	25
13C-2,3,4,7,8-PeCDF	25	25
13C-1,2,3,4,7,8-HxCDD	25	25
13C-1,2,3,4,7,8-HxCDF	25	25
13C-1,2,3,4,7,8,9- HpCDF	25	25

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FIGURE 1: 2378-TCDD CHROMATOGRAPHIC SEPARATION

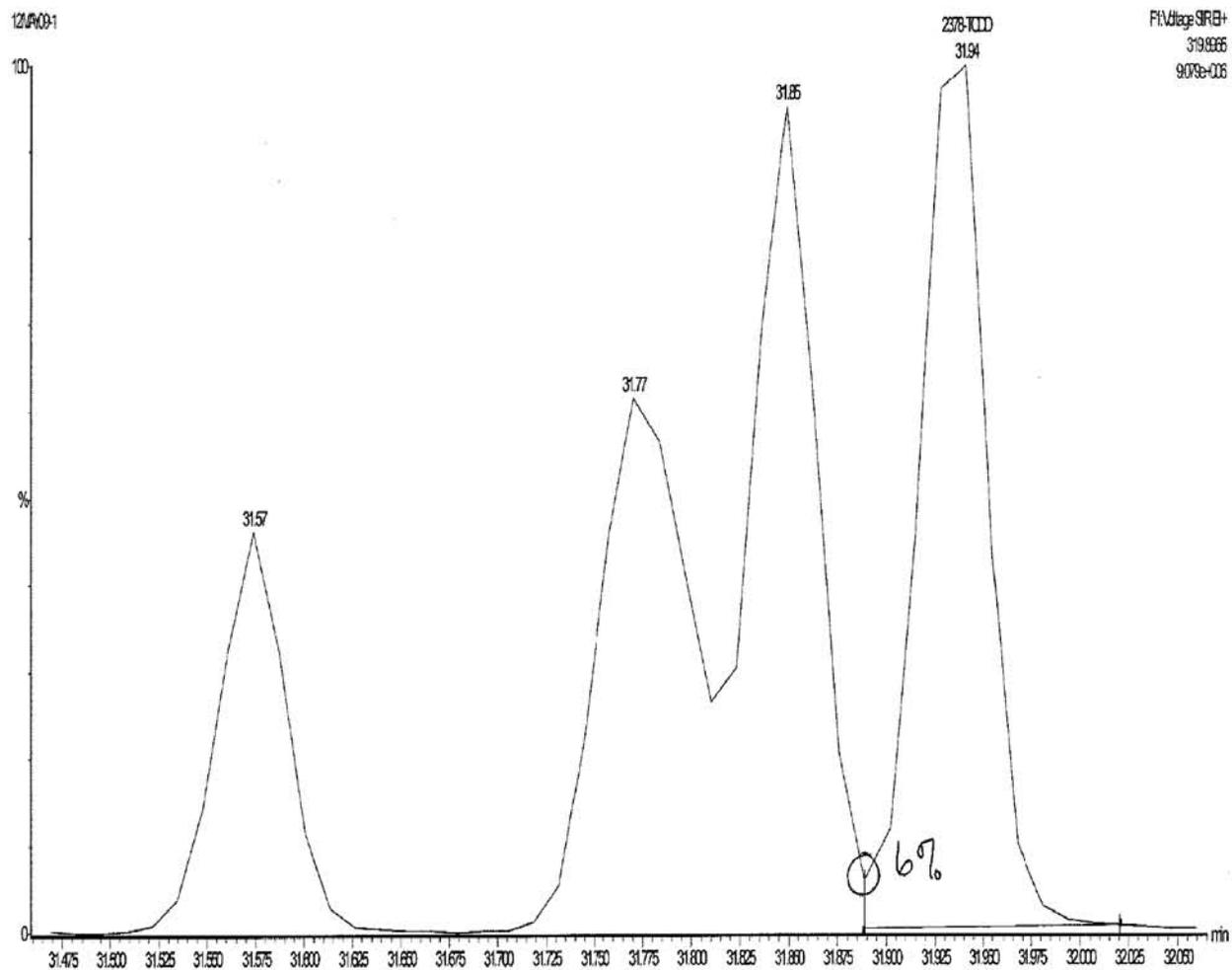
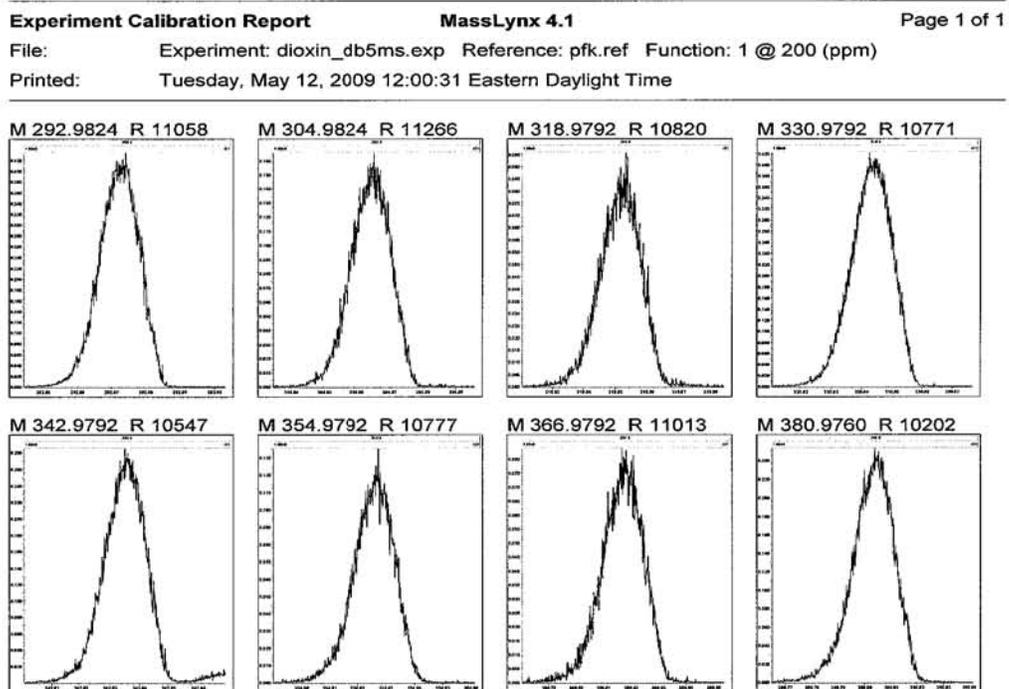


FIGURE 2: INSTRUMENT RESOLVING POWER (EXAMPLE)



VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

SAMPLE RECEIPT, LOGIN, AND STORAGE

(CF-SR-E-001 REVISION 2)

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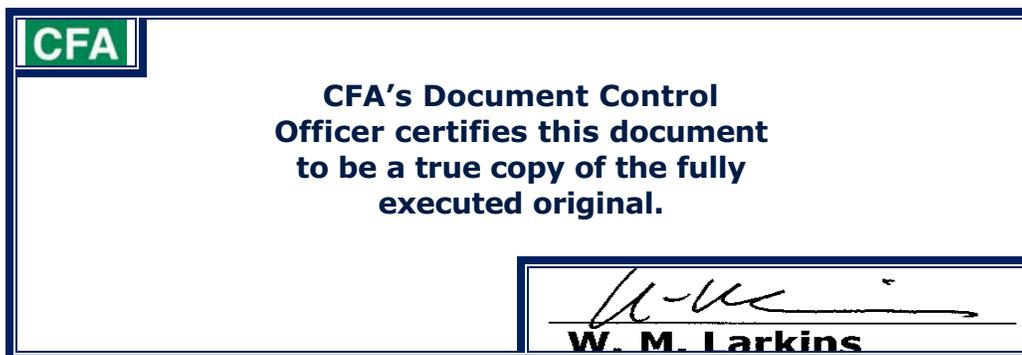


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1.0 STANDARD OPERATING PROCEDURE FOR SAMPLE RECEIPT, LOGIN, AND STORAGE

2.0 PURPOSE

To describe the routine operational procedures for the receipt, login, and storage of samples received by Cape Fear Analytical, LLC (CFA). This SOP also describes how the laboratory will manage relocation of samples in the event of an impending emergency, such as a hurricane.

3.0 DISCUSSION

3.1 Sample custody is a pre-planned mechanism for tracking a sample from the collection of the sample in the field through the release of the finished analytical data to the client. At the collection site, the sample containers are filled with sample and the chain of custody (CoC) form is initiated. The sample collector fills out the form, which includes the name of the client, the requested analysis parameters, sample location, the date and time of collection, sampling technique, preservatives used, and any comments or remarks that may be useful in the analytical work or data interpretation that will follow. Proper sample receipt, login and storage assure accurate chain of custody.

3.2 Custody is defined as:

Being in your physical possession, or

Being in your view, after being in your possession, or

Being locked up after being in your possession, or

Being in a designated secure area

3.3 Upon arrival at the laboratory, sampling personnel, delivery service and carriers relinquish the samples to the sample management group. Each sample container receives a unique sample identifier that is assigned electronically by AlphaLIMS (CFA's Laboratory Information Management System). AlphaLIMS tracks the status and location of each sample container, and serves as the database for analytical results.

4.0 DEFINITIONS

4.1 AlphaLIMS: The Laboratory Information Management System used at CFA.

4.2 Chain of Custody (CoC): A written record of sample transfer and possession.

4.3 Custody Seal: Security seals that are attached to sample containers and/or bottles that are used to detect unauthorized tampering.

4.4 Holding Time: The allowable period of time between sample collection and preparation or analysis (usually method defined).

4.5 Material Safety Data Sheet (MSDS): A document that may accompany samples of known chemical characteristics. (Refer to CFA's Safety, Health and Chemical Hygiene Plan for more information on MSDSs.)

4.6 Matrix: The physical appearance or make-up of a sample (groundwater, drinking water, wastewater, soil, sludge, etc.) as determined by the client or Project Manager.

4.7 Preservative: Additives that are introduced to a sample at the time of collection to help retard chemical and biological changes that may occur.

- 4.8 **Sample:** Any item that has been submitted for analysis to CFA.
- 4.9 **Sample Delivery Group (SDG):** One or more samples (typically not to exceed 20 samples) from a specific client that are reported by the laboratory at the same time.
- 4.10 **Sample Receipt Review (SRR):** A form used to document a sample's arrival and the condition of its arrival at the laboratory.
- 4.11 **Turn Around Time (TAT):** A numeric designation to the degree of attention a sample should receive. This designation is used to convey the client's requested data delivery dates to the laboratory.

5.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

- 5.1 All samples must be handled with care during the login process. Wear protective gear such as gloves, safety glasses and laboratory coats when handling all samples. Some samples may be accompanied by MSDSs that contain vital information on potential hazards. The sample description and client labels may also give this information.
- 5.2 If there is a spill of a known hazard (based on historical results, MSDSs, and/or sample description), immediately contact the Quality Manager as appropriate.
- 5.3 All sample management personnel are required to read and understand CFA's Safety, Health and Chemical Hygiene Plan.

6.0 PROCEDURES

6.1 Sample Package Receipt

- 6.1.1 All sample packages submitted to CFA are received by sample management personnel. Samples are received from a number of carriers including CFA field staff, CFA couriers, individual clients, and public and private shipping companies.

Upon arrival, all sample packages will be inspected for integrity. Note any unusual physical damage, signs of leakage, or evidence that custody seals have been tampered with. If the package appears to be leaking or has any unusual odor, place it under the fume hood and notify the Safety Officer or Project Manager as appropriate before continuing. As appropriate, the client should be contacted for further direction.

- 6.1.3 All discrepancies noted during receipt and inspection shall be recorded using a SRR form in Appendix 2.
 - 6.1.3.1 Client-specific Sample Receipt Review forms may be created by the Project Management Group. These checklists are created because additional sample management comments and checks are required in order to meet quality objectives established for these project samples. Sample Management personnel are responsible for using any client required receipt forms.
- 6.1.4 The CoC should accompany all samples received by the Sample Management Group. The CoC documentation includes sample identification (e.g., MW-1; Lagoon 17; #1234567), sampling date and time, sample collector, requested parameters to be tested, sample location,

preservation type and any special comments. If a sample arrives without this documentation, the Sample Management Group upon receipt can initiate the CoC. Identify this initiation by printing "INITIATED ON RECEIPT" on the CoC form. Alternately, the Project Manager may contact the customer to obtain a chain of custody.

- 6.1.5 Compare the sample labels to the chain of custody, i.e. sample descriptions, collection dates, collection times, number of containers and any other available information. Note any discrepancies between the CoC and samples on the SRR and inform the Project Manager. Sign and date (including time) the CoC in the appropriate box.

NOTE: The labeling system is to be unique and is to include the use of water-resistant labels and indelible ink.

- 6.1.6 Analytical procedure may require preservation of the sample to ensure that changes in the sample's chemistry or biology do not occur. The two predominant preservation techniques used are changing the pH of the sample and cooling the sample to $0^{\circ} \leq 6^{\circ} \text{ C}$. It is important to check and document the holding time, preservation, and temperature of the samples upon arrival to the laboratory. The correct methods of sample storage, chemical preservation, and maximum holding times are shown in Appendix 1. Those samples determined to be nonconforming shall be documented on the SRR and the Project Manager notified.

Verify and document pH preservation using the following procedure:

- 6.1.6.1 Open the container and remove an aliquot of the original sample. Immerse a pH strip into the removed aliquot. When the likelihood that the potential for spilling a sample exists by pouring, a Pasteur pipette should be used to obtain sample to perform the pH check.

- 6.1.6.2 Observe the pH as indicated on the pH strip, and properly discard pH strip and any secondary containers or glassware used in testing.

NOTE: Never use a pH strip that has been contaminated, or one that has already been used.

- 6.1.6.3 Document results of the preservation verification on the appropriate line of the SSR form.

NOTE: If the pH of the sample is determined to be nonconforming, place the sample on hold and notify the Project Manager. The Project Manager will call the client for further direction. If direction is given to adjust the preservation, continue processing the sample and chemically preserve the sample with the appropriate preservative (Appendix 1). Record the lot # of preservative used on the SRR.

- 6.1.6.4 After adding the appropriate preservative to the sample, wait at least 2 minutes and perform steps 6.1.8.1 through 6.1.8.3 again. The preserved sample should now be placed in the appropriate

laboratory sample storage area. Document this on the SRR:
“SAMPLE PRESERVED UPON ARRIVAL.”

NOTE: The preservative verification is made only when the client sample labels indicate either the analytical fraction or the chemical preservative.

- 6.1.6.5 Sample receipt temperature measurement shall be verified through the use of a temperature blank for **each** transport container (such as a cooler) or other sample container measurement when temperature blank is not available. An IR gun or immersion thermometer may be used.
- 6.1.6.5.1 Open the sample cooler.
- 6.1.6.5.2 Remove the Temperature Validation Container (TVC) if provided.
- 6.1.6.5.3 Open the TVC and immerse a thermometer with a valid calibration into the TVC.
- 6.1.6.5.4 Allow the thermometer reading to equilibrate, and read the thermometer result while it is still immersed in the TVC.
- 6.1.6.5.5 Alternately the receipt temperature can be measured with a calibrated infrared temperature (IR) gun by selecting the TVC or another sample within the shipment for receipt/shipping temperature check.
- 6.1.6.5.6 Record the observed reading on the Sample Receipt Review form (Appendix 2), as well as on the CoC if a space is provided: i.e. “TEMP 4° UPON ARRIVAL.”
- 6.1.6.5.7 Temperature verification results of 0 to 6° C are considered conforming for those samples listed as requiring storage at 4° C. The EPA has extended this range from just above the freezing temperature of water to 6° C.
- 6.1.6.5.8 If the initial temperature verification result is determined to be nonconforming, select multiple sample containers (if available) from the shipping container and re-perform the temperature measurements. Document all measurements.
- 6.1.6.5.9 Record the confirmation temperatures on the SRR as well as on the CoC if a space is provided. Label the temperature as a confirmation temperature (i.e., CT = 7.0 °C).
- 6.1.6.5.10 If another container is not available within the shipment to verify the temperature, the secondary

temperature verification is not performed and duly noted.

NOTE: Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples may be considered acceptable if there is evidence that the chilling process has begun (such as arrival on ice).

6.1.6.6 Samples submitted for dioxin/furan or PCB congener analysis should be checked for the presence of residual chlorine at the time of sample receipt. Residual chlorine is checked by using the following procedure:

6.1.6.6.1 Pour an aliquot of the sample into a secondary container. Immerse a potassium iodide/starch paper strip into the secondary container to test the sample. The presence of residual chlorine may be alternatively determined by removing a very small aliquot of the sample using disposable glassware. The aliquot is tested using potassium iodide/starch paper.

6.1.6.6.2 A blue color on the starch paper indicates the presence of residual chlorine. Discard the test strip and any secondary container or glassware used in testing.

6.1.6.7 If residual chlorine is present, document this on the SRR. The sample then requires the addition of 80 mg sodium thiosulfate, as specified by the analytical methods.

6.1.6.8 Solid samples submitted for volatile analysis that are collected in "EnCore" containers shall be delivered to the volatiles laboratory immediately after unpacking to help ensure preparation hold times are met.

6.1.7 A copy of the chain of custody may be printed (colored paper may be used as a practice to identify originals vs. copies) to be retained with the samples in the sample receipt area. The completed original CoC and SRR are delivered to the appropriate Project Manager for pre-logging of the samples.

6.1.8 The Project Manager pre-logs the data from the CoC and SRR into AlphaLIMS. Once samples are pre-logged into the system, the data are verified, and the samples are logged in officially. Unique bar code labels are generated for each sample container upon completion of the log-in. Details of the Project Management process for pre-log and login may be found in CF-CS-M-001 and CF-CS-E-008.

6.1.9 The bar code labels are ready to be affixed to the appropriate containers.

6.1.10 Sample bar code labels are color-coded as follows:

6.1.10.1 Orange for material requiring special waste handling.

6.1.10.2 White for all other samples.

- 6.1.11 Compare the sample description on the printed CFA bar code label to the client sample bottle label before attaching labels to containers. Wherever possible, the CFA label should not cover the client's label or any other information provided by the client or sample collector.
- 6.1.12 If the sample is a solid submitted for volatiles or TCLP VOA analysis and a single container is provided, a designation is generated on the barcode label indicating, "Volatiles must aliquot sample first." It is then stored in the appropriate storage location until removed for volatiles or TCLP volatiles testing. Once the volatiles lab takes its required aliquot the container will be marked with the analyst's initials and the date completed. The sample container will then be placed in the appropriate walk-in cooler and released for other laboratory analyses. Note exception in Section 6.2.2.1.
- 6.1.13 Situations may occur that delay the login process of samples. Examples include: awaiting client direction in response to a nonconformance, discrepancies between chain of custody and sample label information, broken or damaged containers, etc. At the end of each day, an assigned Project Manager will identify and verify any unlabeled sample containers. This inspection will be documented by email to members of the Project Management Group and the Client Services Manager. Samples requiring cold preservation will be placed into refrigerated storage until the labeling issues are resolved.
- 6.1.14 Project Managers and the Client Services Manager will be notified daily of any containers remaining on hold to include relogs, subcontractor samples, or any other sample containers placed in the temporary storage location. This process will be repeated daily until the sample issue is resolved and the sample containers in question are either relocated to proper storage locations or properly packed for shipment.
- 6.2 Sample Storage
- 6.2.1 Once the samples have been properly labeled, the samples are scanned into the electronic tracking system in AlphaLIMS. The samples are placed in the appropriate storage areas located within the laboratory a secure facility with limited access. Refer to CF-LB-E-012 for Verifying the Maintenance of Sample Integrity.
- NOTE:** Samples requiring immediate analysis or those with very quick turn around times (TAT) may be made available to the laboratory prior to label application. In these cases, the label is applied to any remaining sample container after the sample has been made available for analysis. This sample type includes VOA EnCores which require immediate preservation by the VOA group.
- 6.2.2 Samples are placed in numerical order in the appropriate storage locations throughout the facility. Containers are 'loaded' into the system by container type and size (i.e., 1000 mL Nalgene), preservative (i.e., sulfuric acid), and storage area destination.

6.2.2.1 Samples requiring analysis of volatile organics shall be segregated from other samples by placing them in either the Volatiles cooler, which is located in the Volatiles area and maintained at $0 \leq 6$ °C. EnCore samples are placed in the designated freezer. EnCore kits containing 40 mL vials are placed in the freezer at a slight tilt or a 45° angle to prevent breakage due to the expansion of liquid.

NOTE: Samples requiring volatile analyses known to contain high concentrations of organic solvents or hydrocarbons should not be stored in the volatiles coolers. Place these samples in the general use walk-in cooler.

6.2.2.2 Samples requiring cold preservation (other than volatile organics samples) are stored in numerical order in general use walk-in coolers, which are maintained at $0 \leq 6$ °C.

6.2.3 Coolers are monitored in accordance with CF-LB-E-004, Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens and Other Similar Devices, for requirements associated with temperature monitoring and temperature monitoring devices.

6.3 Sample Shipment During Impending Emergencies

6.3.1 In the event of an impending emergency (such as a hurricane), it may be necessary to move samples out of the laboratory to an appropriate temporary storage location. A subcontractor laboratory or other storage facility which meets the requirements for proper sample storage (as discussed above) will be identified by the Quality Manager. Chain of Custody documentation and proper storage conditions for all samples and their extracts must be available for temporary storage situations.

6.4 Project Manager Notification and Action

6.4.1 Lab personnel are required to notify the project manager of any anomalies detected during the login process.

6.4.2 A member of project management will attempt to contact the client and resolve any issues with sample acceptance. It may be possible to continue with analysis with client permission, although the data may be qualified.

6.4.3 Project management will clarify the client's wishes to the laboratory and monitor the laboratory's performance in relation to the client's desired outcome.

7.0 RECORDS MANAGEMENT

7.1 The Sample Receipt Review form is attached to the chain of custody and forwarded to the Project Manager.

7.2 Cooler temperature logs are reviewed in AlphaLIMS. Refer to CF-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, and Other Similar Devices.

8.0 REFERENCES

Example Standard Operating Procedures for Contract Laboratory Program (CLP), National Enforcement Investigations Center (NEIC), Contract Evidence Audit Team (CEAT-TechLaw), EPA Contract 68-01-6838, 1986.

9.0 HISTORY

Revision 0: New document.

Revision 1: Subcontracting the storage of samples/extracts in an emergency.

Revision 2: PM notification and actions detailed. Sample acceptance policy added.

APPENDIX 1: STORAGE AND PRESERVATION**SAMPLE STORAGE AND PRESERVATION REQUIREMENTS**

Parameter	Container¹	Preservation	Holding Time²	Min. Volume
<u>Organics</u>				
PCB Congeners	Amber G, teflon-lined cap	0 ≤ 6° C; <-10°C for tissues; 80 mg sodium thiosulfate ³	1 year for extraction; 45 days after extraction for analysis	1000 mL / 50 g
Dioxin/Furan	Amber G, teflon-lined cap	0 ≤ 6° C; <-10°C for tissues; 80 mg sodium thiosulfate ³	30 days (8290) or 1 year (1613) for extraction; 45 days after extraction for analysis	1000 mL / 50 g
Formaldehyde	Amber G, teflon-lined cap	0 ≤ 6° C	3 days for derivitization/extraction; 3 days after extraction for analysis	1000 mL / 50 g

¹ P = Polyethylene; G = Glass² Samples should be analyzed as soon as possible after collection. The holding times listed are maximum times that samples may be held before analysis and be considered valid.³ Used only in the presence of residual chlorine in water samples.

Sample Receipt, Login, and Storage

SOP Effective 04/14/09

CF-SR-E-001 Rev 2

Revision 2 Effective Aug 2010 Last reviewed Aug 2010

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APPENDIX 2: SAMPLE RECEIPT REVIEW FORM

SAMPLE RECEIPT & REVIEW FORM

Client:	Work Order:
Received By:	Date Received:

Suspected Hazard Information	Yes	NA	No
Shipped as DOT Hazardous?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Samples identified as Foreign Soil?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

#	Sample Receipt Criteria	Yes	NA	No	Comments/Qualifiers (required for Non-Conforming Items)
1	Shipping containers received intact and sealed?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Circle Applicable: seals broken damaged container leaking container other(describe)
2	Samples requiring cold preservation within 0-6°C?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Preservation Method: ice bags blue ice dry ice none other (describe)
3	Chain of Custody documents included with shipment?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
4	Sample containers intact and sealed?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Circle Applicable: seals broken damaged container leaking container other(describe)
5	Samples requiring chemical preservation at proper pH?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Sample IDs, containers affected and pH observed: If preservative added, Lot#:
6	VOA vials free of headspace <6mm bubble?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Sample IDs, containers affected:
7	Are Encore containers present?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	(If YES, immediately deliver to volatiles laboratory)
8	Samples received within holding time?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Sample IDs, tests affected:
9	Sample IDs on COC match IDs on containers?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Sample IDs, containers affected:
10	Date & time of COC match date & time on containers?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Sample IDs, containers affected:
11	Number of containers received match number indicated on COC?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Sample IDs, containers affected:
12	COC form is properly signed in relinquished/received sections?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	

Comments:

PM review: Initials: _____ Date: _____

APPENDIX 3: SAMPLE ACCEPTANCE POLICY**Sample Acceptance Policy***Cape Fear Analytical, LLC.*

Samples received by the laboratory must meet certain criteria in order for the associated analytical data to meet method criteria. The validity and defensibility of analytical data is highly dependent upon proper delivery of samples to the laboratory. The following criteria are used to evaluate samples prior to their acceptance for analysis:

a) Documentation: All samples must be accompanied by chain-of-custody forms indicating unique sample identification, date and time of sampling, location of collection, collector's name, preservation type, sample type (e.g. soil, water, etc.), requested analyses and any special remarks concerning the samples.

b) Sample Labeling: Each sample must be given a unique identifier that allows for cross-referencing with the chain-of-custody information. Sample labels must be durable enough to remain attached to the containers and utilize indelible ink to remain legible during shipping.

c) Sample Containers: Samples must be shipped in containers appropriate for the required analyses. Liquid and solid samples collected for organics analyses should be shipped in glass containers. Project management is available to answer any questions regarding sample containers (prior to collection). Containers should be packaged in such a way as to prevent breakage or cross contamination during shipping.

d) Holding Times: Most analytical methods provide limits for the amount of time that may elapse between collection, extraction, and analysis of samples. Data from the analysis of a sample that have exceeded its required holding time is generally considered invalid and is not legally defensible. Project management will contact clients when samples are received near their holding time expiration. Such samples may not be accepted by the laboratory.

e) Sample Volume: Sufficient sample volume must arrive intact for all required tests. Each method specifies the required sample volume for each applicable matrix. Project management is available to the client to answer questions regarding required sample volumes (prior to sampling).

f) Samples that are immediately **rejected** are ones with **improper preservation, damage**, or show signs of **contamination**. A member of project management will attempt to contact the client and resolve any issues with sample acceptance. It may be possible to continue with analysis with client permission, although the data may be qualified. The Technical Director is available to project management and the client to describe the problem and any potential effects on the data. The Technical Director will make the final decision regarding the laboratory's acceptance of samples.

Damage: Samples are inspected for signs of damage - Damaged containers must be safely repackaged and quarantined. Record the damage found and action taken.

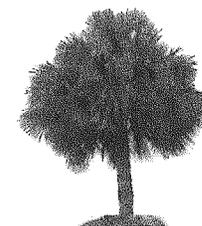
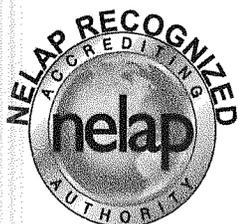
Contamination: The shipment is inspected for signs of contamination - Separate the samples and quarantine any further means of contamination. Record the condition of the samples and action taken.

Preservation: It is generally required that samples be chilled to < 6°C and kept at that temperature during shipping. Therefore, samples should be placed in insulated coolers with ice to keep them at or near this temperature until delivery at the laboratory. Samples that arrive above 6°C and are not on ice will be rejected and the client will be contacted for further instructions, such as re-sampling if for compliance monitoring. Discrepancy Procedure: Inadequate preservation must be reported to client services.

g) Client Notification: When sample login is completed, a summary of the sample receipt and product assignment is generated. This can be delivered to the client upon request.

3306 Kitty Hawk Road Suite 120 Wilmington, NC 28405 (910) 795-0421

Katahdin SOPs and Accreditations



State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E87604

KATAHDIN ANALYTICAL SERVICES, INC.
600 TECHNOLOGY WAY
SCARBOROUGH, ME 04074

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - RADIOCHEMISTRY, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

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.

EFFECTIVE July 01, 2010 THROUGH June 30, 2011



Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04

NON-TRANSFERABLE E87604-15-07/01/2010
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/26/2002
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Hexanone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Acetone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Alkalinity as CaCO ₃	SM 2320 B	Primary Inorganic Contaminants	NELAP	4/26/2002
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/26/2002
Amenable cyanide	SM 4500-CN G	Primary Inorganic Contaminants	NELAP	2/4/2002
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/26/2002
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Bromoform	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Calcium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Carbon disulfide	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	4/26/2002
Chloride	EPA 325.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Chloroform	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Color	EPA 110.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	4/17/2007
Copper	EPA 200.7	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Copper	EPA 200.8	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	2/4/2002
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Diethyl ether	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Escherichia coli	SM 9223 B	Microbiology	NELAP	3/22/2010
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Heterotrophic plate count	SIMPLATE	Microbiology	NELAP	11/7/2006
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Iron	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Magnesium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2004
Perchlorate	EPA 314.0	Secondary Inorganic Contaminants	NELAP	7/30/2004
pH	EPA 150.1	Primary Inorganic Contaminants, Secondary Inorganic Contaminants	NELAP	2/4/2002

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Scarborough, ME 04074

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
pH	SM 4500-H+-B	Secondary Inorganic Contaminants	NELAP	4/17/2007
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Silica as SiO ₂	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Sodium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Sulfate	ASTM D516-02	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	ASTM D516-90	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	EPA 300.0	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Tetrahydrofuran (THF)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Total coliforms	SM 9223 B	Microbiology	NELAP	3/22/2010
Total dissolved solids	EPA 160.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Total organic carbon	SM 5310 B	Primary Inorganic Contaminants	NELAP	5/8/2009
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Uranium	EPA 200.8	Radiochemistry	NELAP	11/7/2006
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4'-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	2/4/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	Semivolatiles/GC-MS			
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	Semivolatiles/GC-MS			
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
Acenaphthene	Semivolatiles/GC-MS			
Acenaphthylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	Semivolatiles/GC-MS			
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
Acetone	Low-Medium			
Acetone	Volatiles/GC-MS			
Acetone	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
Acetone	Volatiles/GC-MS			
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acetophenone	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
Acetophenone	Semivolatiles/GC-MS			
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	2/4/2002
Acidity, as CaCO3	SM 2310 B (4A)	General Chemistry	NELAP	4/17/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	4/26/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	4/26/2002

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	2/4/2002
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	7/30/2004
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	2/4/2002
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 200.7	Metals	NELAP	2/4/2002
Aluminum	EPA 200.8	Metals	NELAP	2/4/2002
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aluminum	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	2/4/2002
Ammonia as N	EPA 350.1	General Chemistry	NELAP	2/4/2002
Ammonia as N	SM 4500-NH3 H	General Chemistry	NELAP	2/4/2002
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 200.7	Metals	NELAP	2/4/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Antimony	EPA 200.8	Metals	NELAP	2/4/2002
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Antimony	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 200.7	Metals	NELAP	2/4/2002
Arsenic	EPA 200.8	Metals	NELAP	2/4/2002
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Arsenic	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Atrazine	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 200.7	Metals	NELAP	2/4/2002
Barium	EPA 200.8	Metals	NELAP	2/4/2002
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Barium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002

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Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	2/4/2002
Beryllium	EPA 200.8	Metals	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Beryllium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	2/4/2002
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	2/4/2002
Biphenyl	EPA 8270	Extractable Organics	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	2/4/2002

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 200.7	Metals	NELAP	2/4/2002
Boron	EPA 6010	Metals	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	2/4/2002
Bromide	EPA 9056	General Chemistry	NELAP	7/1/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Charlie Crist
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Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 200.7	Metals	NELAP	2/4/2002
Cadmium	EPA 200.8	Metals	NELAP	2/4/2002
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cadmium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Calcium	EPA 200.7	Metals	NELAP	2/4/2002
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Calcium	EPA 6020	Metals	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	2/4/2002

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State Surgeon General

Laboratory Scope of Accreditation

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600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/26/2002
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	2/4/2002
Chemical oxygen demand	HACH 8000	General Chemistry	NELAP	4/26/2002
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	2/4/2002
Chloride	EPA 325.2	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	7/1/2003
Chloride	EPA 9251	General Chemistry	NELAP	7/1/2003
Chloride	SM 4500 Cl- E	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	2/4/2002
Chromium	EPA 200.8	Metals	NELAP	4/26/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	4/26/2002
Chromium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Chromium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 625	Extractable Organics	NELAP	2/4/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cobalt	EPA 200.7	Metals	NELAP	2/4/2002
Cobalt	EPA 200.8	Metals	NELAP	2/4/2002
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cobalt	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Color	EPA 110.2	General Chemistry	NELAP	4/26/2002
Color	SM 2120 B	General Chemistry	NELAP	2/4/2002
Conductivity	EPA 120.1	General Chemistry	NELAP	2/4/2002
Conductivity	SM 2510 B	General Chemistry	NELAP	2/4/2002
Copper	EPA 200.7	Metals	NELAP	2/4/2002
Copper	EPA 200.8	Metals	NELAP	2/4/2002

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Cyanide	EPA 335.3	General Chemistry	NELAP	2/4/2002
Cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Decachlorobiphenyl (BZ 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenzofuran	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002

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600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	7/1/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Escherichia coli	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	9/4/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	5/12/2005

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9222 D	Microbiology	NELAP	7/30/2004
Fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluoride	EPA 340.2	General Chemistry	NELAP	2/4/2002
Fluoride	SM 4500 F-C	General Chemistry	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	7/1/2003
Hardness	SM 2340 B	Metals	NELAP	2/4/2002
Hardness (calc.)	EPA 200.7	Metals	NELAP	4/26/2002
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	EPA 6020	Metals	NELAP	11/7/2006
Iron	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Iron	SM 3500-Fe D (18th/19th Ed.)UV-VIS	General Chemistry	NELAP	4/26/2002
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Extractable Organics	NELAP	7/1/2003

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600 Technology Way

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Isophorone	EPA 625	Extractable Organics	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	2/4/2002
Lead	EPA 200.7	Metals	NELAP	2/4/2002
Lead	EPA 200.8	Metals	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Lead	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 200.7	Metals	NELAP	2/4/2002
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Magnesium	EPA 6020	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Manganese	EPA 200.7	Metals	NELAP	2/4/2002
Manganese	EPA 200.8	Metals	NELAP	2/4/2002
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	4/26/2002
Mercury	EPA 245.1	Metals	NELAP	2/4/2002
Mercury	EPA 7470	Metals	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylene chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
Methylene chloride	Low-Medium Volatiles/GC-MS			
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 200.7	Metals	NELAP	2/4/2002
Molybdenum	EPA 200.8	Metals	NELAP	2/4/2002
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Molybdenum	EPA 6020	Metals	NELAP	4/26/2002
Naphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	2/4/2002
Nickel	EPA 200.8	Metals	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nickel	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrite as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrite as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrite as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodiphenylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	2/4/2002
Oil & Grease	EPA 9070	General Chemistry	NELAP	7/1/2003
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 365.1	General Chemistry	NELAP	11/7/2006
Orthophosphate as P	EPA 365.2	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	7/1/2003
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003

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E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 150.1	General Chemistry	NELAP	2/4/2002
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	4/26/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	2/4/2002
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	7/1/2003
Potassium	EPA 6020	Metals	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	2/4/2002
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	2/4/2002
Residue-settleable	EPA 160.5	General Chemistry	NELAP	2/4/2002
Residue-settleable	SM 2540 F	General Chemistry	NELAP	2/4/2002
Residue-total	EPA 160.3	General Chemistry	NELAP	2/4/2002
Residue-total	SM 2540 B	General Chemistry	NELAP	2/4/2002
Residue-volatile	EPA 160.4	General Chemistry	NELAP	2/4/2002
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Salinity	SM 2520 B	General Chemistry	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	2/4/2002
Selenium	EPA 200.8	Metals	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Selenium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silicon	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.8	Metals	NELAP	2/4/2002
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	2/4/2002
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	EPA 6020	Metals	NELAP	11/7/2006

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	ASTM D516-02	General Chemistry	NELAP	4/17/2007
Sulfate	ASTM D516-90	General Chemistry	NELAP	4/17/2007
Sulfate	EPA 300.0	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 375.4	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9038	General Chemistry	NELAP	7/1/2003
Sulfate	EPA 9056	General Chemistry	NELAP	7/1/2003
Sulfide	EPA 376.1	General Chemistry	NELAP	2/4/2002
Sulfide	SM 4500-S E (18th Ed.)/TITR	General Chemistry	NELAP	4/17/2007
Sulfite-SO3	EPA 377.1	General Chemistry	NELAP	2/4/2002
Sulfite-SO3	SM 4500-SO3 B	General Chemistry	NELAP	4/17/2007
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	5/12/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 200.7	Metals	NELAP	2/4/2002
Thallium	EPA 200.8	Metals	NELAP	2/4/2002
Thallium	EPA 6010	Metals	NELAP	7/1/2003

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thallium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Thorium	EPA 200.8	Metals	NELAP	2/4/2002
Tin	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Tin	EPA 200.7	Metals	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 200.7	Metals	NELAP	2/4/2002
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 624	Volatile Organics	NELAP	2/4/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total coliforms	SM 9222 B	Microbiology	NELAP	7/30/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Total hardness as CaCO ₃	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Total hardness as CaCO ₃	EPA 130.2	General Chemistry	NELAP	9/4/2007
Total hardness as CaCO ₃	SM 2340 C	General Chemistry	NELAP	9/4/2007
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Total organic carbon	EPA 415.1	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic carbon	SM 5310 B	General Chemistry	NELAP	4/17/2007
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 420.1	General Chemistry	NELAP	2/4/2002
Total phenolics	EPA 9065	General Chemistry	NELAP	7/1/2003
Total residual chlorine	SM 4500-Cl G	General Chemistry	NELAP	9/4/2007
Total, fixed, and volatile residue	SM 2540 G	General Chemistry	NELAP	4/26/2002
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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EPA Lab Code: ME00019

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Turbidity	EPA 180.1	General Chemistry	NELAP	2/4/2002
Turbidity	SM 2130 B	General Chemistry	NELAP	2/4/2002
Uranium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 200.7	Metals	NELAP	2/4/2002
Vanadium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 6010	Metals	NELAP	7/1/2003

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vanadium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/26/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 200.7	Metals	NELAP	2/4/2002
Zinc	EPA 200.8	Metals	NELAP	2/4/2002
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	2/4/2002
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Zinc	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006

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Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,1-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,1-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002

Laboratory Scope of Accreditation

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Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	4/26/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	4/26/2002
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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State Surgeon General

Laboratory Scope of Accreditation

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4'-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	4/26/2002
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Scarborough, ME 04074**

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Analyte	Method/Tech	Category	Certification Type	Effective Date
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

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600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acetone	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetophenone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	4/26/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 6010	Metals	NELAP	2/4/2002
Aluminum	EPA 6020	Metals	NELAP	2/4/2002
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Aniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 6010	Metals	NELAP	2/4/2002
Antimony	EPA 6020	Metals	NELAP	2/4/2002
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 6010	Metals	NELAP	2/4/2002
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Atrazine	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 6010	Metals	NELAP	2/4/2002
Barium	EPA 6020	Metals	NELAP	2/4/2002
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	2/4/2002
Beryllium	EPA 6020	Metals	NELAP	2/4/2002
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 6010	Metals	NELAP	4/26/2002
Bromide	EPA 9056	General Chemistry	NELAP	2/4/2002
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 6010	Metals	NELAP	2/4/2002
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Calcium	EPA 6010	Metals	NELAP	2/4/2002
Calcium	EPA 6020	General Chemistry	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9251	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chromium	EPA 6010	Metals	NELAP	2/4/2002
Chromium	EPA 6020	Metals	NELAP	2/4/2002
Chromium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium VI	EPA 7196	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	Semivolatiles/GC-MS			
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
	Low-Medium			
	Volatiles/GC-MS			
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
	Volatiles/GC-MS			
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
	Low-Medium			
	Volatiles/GC-MS			
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
	Volatiles/GC-MS			
Cobalt	EPA 6010	Metals	NELAP	2/4/2002
Cobalt	EPA 6020	Metals	NELAP	2/4/2002
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	EPA 6010	Metals	NELAP	2/4/2002
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
	Low-Medium			
	Volatiles/GC-MS			
Cyclohexane	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
	Volatiles/GC-MS			
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Decachlorobiphenyl (BZ 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

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600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	2/4/2002
Diethyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Laboratory Scope of Accreditation

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
	Semivolatiles/GC-MS			
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
	Pesticides/GC-ECD			
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
	Low-Medium Volatiles/GC-MS			
Ethylbenzene	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
	Volatiles/GC-MS			
Ethyl-t-butylether (ETBE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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EPA Lab Code:

ME00019

(207) 874-2400

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Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Ignitability	EPA 1010	General Chemistry	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	2/4/2002
Iron	EPA 6020	General Chemistry	NELAP	11/7/2006
Iron	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	2/4/2002
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 6010	Metals	NELAP	2/4/2002
Magnesium	EPA 6020	General Chemistry	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Manganese	EPA 6010	Metals	NELAP	2/4/2002

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Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

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EPA Lab Code: ME00019

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E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Manganese	EPA 6020	Metals	NELAP	2/4/2002
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Mercury	EPA 7471	Metals	NELAP	2/4/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methapyrilene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

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State Surgeon General

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EPA Lab Code: ME00019

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600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 6010	Metals	NELAP	4/26/2002
Molybdenum	EPA 6020	General Chemistry	NELAP	11/7/2006
Naphthalene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Naphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	2/4/2002
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	4/26/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002

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State Surgeon General

Laboratory Scope of Accreditation

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Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 9071	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	2/4/2002
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
p-Dioxane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 9040	General Chemistry	NELAP	2/4/2002
pH	EPA 9045	General Chemistry	NELAP	2/4/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	2/4/2002
Potassium	EPA 6020	General Chemistry	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	2/4/2002
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	2/4/2002
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Safrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	2/4/2002
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	EPA 6010	Metals	NELAP	2/4/2002
Silver	EPA 6020	Metals	NELAP	2/4/2002
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Sodium	EPA 6010	Metals	NELAP	2/4/2002
Sodium	EPA 6020	General Chemistry	NELAP	11/7/2006
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	2/4/2002
Styrene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	EPA 9038	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9056	General Chemistry	NELAP	2/4/2002
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	2/4/2002
T-amylmethylether (TAME)	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 6010	Metals	NELAP	2/4/2002
Thallium	EPA 6020	Metals	NELAP	2/4/2002
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 9065	General Chemistry	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	4/26/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vanadium	EPA 6010	Metals	NELAP	2/4/2002
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 6010	Metals	NELAP	2/4/2002
Zinc	EPA 6020	Metals	NELAP	2/4/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Mercury	EPA 7470	General Chemistry	NELAP	7/30/2004
Mercury	EPA 7471	General Chemistry	NELAP	7/30/2004
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004



**Scope of Accreditation
For
Katahdin Analytical Services, Inc.**

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

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 Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 608 / 8081B	4,4'-DDD
GC/ECD	EPA 608 / 8081B	4,4'-DDE
GC/ECD	EPA 608 / 8081B	4,4'-DDT
GC/ECD	EPA 608 / 8081B	Aldrin
GC/ECD	EPA 608 / 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 608 / 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608 / 8081B	Chlordane (tech.)
GC/ECD	EPA 608 / 8081B	delta-BHC
GC/ECD	EPA 608 / 8081B	Dieldrin
GC/ECD	EPA 608 / 8081B	Endosulfan I
GC/ECD	EPA 608 / 8081B	Endosulfan II
GC/ECD	EPA 608 / 8081B	Endosulfan sulfate
GC/ECD	EPA 608 / 8081B	Endrin
GC/ECD	EPA 608 / 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608 / 8081B	Heptachlor
GC/ECD	EPA 608 / 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 608 / 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 608 / 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608 / 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608 / 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608 / 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608 / 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608 / 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608 / 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4', 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)



Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPD
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015B/C MOD	Diesel range organics (DRO)
GC/FID	EPA 8015B/C MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011 / 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011 / 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B,C / 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1, 2-Trichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1-Dichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B,C / 524.2	1, 1-Dichloropropene
GC/MS	EPA 8260B,C / 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B,C / 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B,C / 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C / 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B,C / 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 624 / 8260B,C / 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B,C / 524.2	1, 3-Dichloropropane
GC/MS	EPA 624 / 8260B,C / 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 4-Dioxane
GC/MS	EPA 8260B,C / 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B,C / 524.2	2-Butanone
GC/MS	EPA 624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B,C / 524.2	2-Chlorotoluene
GC/MS	EPA 8260B,C / 524.2	2-Hexanone
GC/MS	EPA 8260B,C / 524.2	4-Chlorotoluene
GC/MS	EPA 8260B,C / 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B,C / 524.2	Acetone
GC/MS	EPA 8260B,C	Acetonitrile
GC/MS	EPA 1624 / 8260B,C	Acrolein
GC/MS	EPA 624 / 8260B,C / 524.2	Acrylonitrile



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B,C / 524.2	Allyl chloride
GC/MS	EPA 624 / 8260B,C / 524.2	Benzene
GC/MS	EPA 8260B,C / 524.2	Bromobenzene
GC/MS	EPA 8260B,C / 524.2	Bromochloromethane
GC/MS	EPA 624 / 8260B,C / 524.2	Bromodichloromethane
GC/MS	EPA 624 / 8260B,C / 524.2	Bromoform
GC/MS	EPA 8260B,C / 524.2	Carbon disulfide
GC/MS	EPA 624 / 8260B,C / 524.2	Carbon tetrachloride
GC/MS	EPA 624 / 8260B,C / 524.2	Chlorobenzene
GC/MS	EPA 624 / 8260B,C / 524.2	Chloroethane
GC/MS	EPA 624 / 8260B,C / 524.2	Chloroform
GC/MS	EPA 8260B,C	Chloroprene
GC/MS	EPA 8260B,C / 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 624 / 8260B,C / 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B,C	Cyclohexane
GC/MS	EPA 624 / 8260B,C / 524.2	Dibromochloromethane
GC/MS	EPA 8260B,C / 524.2	Dibromomethane
GC/MS	EPA 624 / 8260B,C / 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B,C / 524.2	Diethyl ether
GC/MS	EPA 8260B,C	Di-isopropylether
GC/MS	EPA 8260B,C / 524.2	Ethyl methacrylate
GC/MS	EPA 624 / 8260B,C / 524.2	Ethylbenzene
GC/MS	EPA 8260B,C	Ethyl-t-butylether
GC/MS	EPA 8260B,C / 524.2	Hexachlorobutadiene
GC/MS	EPA 8260B,C	Iodomethane
GC/MS	EPA 8260B,C	Isobutyl alcohol
GC/MS	EPA 8260B,C / 524.2	Isopropyl benzene
GC/MS	EPA 8260B,C / 524.2	m p-xylenes
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B,C / 524.2	Methacrylonitrile
GC/MS	EPA 624 / 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 624 / 8260B,C / 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B,C / 524.2	Methyl methacrylate

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B,C / 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B,C	Methylcyclohexane
GC/MS	EPA 624 / 8260B,C / 524.2	Methylene chloride
GC/MS	EPA 8260B,C / 524.2	Naphthalene
GC/MS	EPA 8260B,C / 524.2	n-Butylbenzene
GC/MS	EPA 8260B,C / 524.2	n-Propylbenzene
GC/MS	EPA 8260B,C / 524.2	o-Xylene
GC/MS	EPA 8260B,C / 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B,C / 524.2	Propionitrile
GC/MS	EPA 8260B,C / 524.2	sec-butylbenzene
GC/MS	EPA 8260B,C / 524.2	Styrene
GC/MS	EPA 8260B,C	t-Amylmethylether
GC/MS	EPA 8260B,C / 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B,C	tert-Butylbenzene
GC/MS	EPA 624 / 8260B,C / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B,C / 524.2	Tetrahydrofuran
GC/MS	EPA 624 / 8260B,C / 524.2	Toluene
GC/MS	EPA 624 / 8260B,C / 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 624 / 8260B,C / 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B,C / 524.2	trans-1, 4-Dichloro-2-butene
GC/MS	EPA 624 / 8260B,C / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624 / 8260B,C / 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B,C	Vinyl acetate
GC/MS	EPA 624 / 8260B,C / 524.2	Vinyl chloride
GC/MS	EPA 624 / 8260B,C	Xylene
GC/MS	EPA 8270C,D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 625 / 8270C,D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 625 / 8270C,D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C,D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 625 / 8270C,D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 3-Dinitrobenzene
GC/MS	EPA 625 / 8270C,D	1, 4-Dichlorobenzene



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	1, 4-Dioxane
GC/MS	EPA 8270C,D	1, 4-Naphthoquinone
GC/MS	EPA 8270C,D	1, 4-Phenylenediamine
GC/MS	EPA 8270C,D	1-Naphthylamine
GC/MS	EPA 8270C,D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C,D	2, 4, 5-Trochlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4, 6-Trichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dimethylphenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dinitrophenol
GC/MS	EPA 625 / 8270C,D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C,D	2, 6-Dichlorophenol
GC/MS	EPA 625 / 8270C,D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C,D	2-Acetylaminofluorene
GC/MS	EPA 625 / 8270C,D	2-Chloronaphthalene
GC/MS	EPA 625 / 8270C,D	2-Chlorophenol
GC/MS	EPA 625 / 8270C,D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C,D	2-Methylnaphthalene
GC/MS	EPA 8270C,D	2-Methylphenol
GC/MS	EPA 8270C,D	2-Naphthylamine
GC/MS	EPA 8270C,D	2-Nitroaniline
GC/MS	EPA 625 / 8270C,D	2-Nitrophenol
GC/MS	EPA 8270C,D	2-Picoline
GC/MS	EPA 625 / 8270C,D	3, 3'-Dichlorobenzidine
GC/MS	EPA 8270C,D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C,D	3-Methylcholanthrene
GC/MS	EPA 8270C,D	3-Nitroaniline
GC/MS	EPA 8270C,D	4-Aminobiphenyl
GC/MS	EPA 625 / 8270C,D	4-Bromophenyl phenyl ether
GC/MS	EPA 625 / 8270C,D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C,D	4-Chloroaniline
GC/MS	EPA 625 / 8270C,D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C,D	4-Dimethyl aminoazobenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	4-Methylphenol
GC/MS	EPA 8270C,D	4-Nitroaniline
GC/MS	EPA 625 / 8270C,D	4-Nitrophenol
GC/MS	EPA 8270C,D	5-Nitro-o-toluidine
GC/MS	EPA 8270C,D	7,12-Dimethylphenethylamine
GC/MS	EPA 8270C,D	a a-Dimethylphenethylamine
GC/MS	EPA 625 / 8270C,D	Acenaphthene
GC/MS	EPA 625 / 8270C,D	Acenaphthylene
GC/MS	EPA 8270C,D	Acetophenone
GC/MS	EPA 8270C,D	Aniline
GC/MS	EPA 625 / 8270C,D	Anthracene
GC/MS	EPA 8270C,D	Aramite
GC/MS	EPA 8270C,D	Atrazine
GC/MS	EPA 625 / 8270C,D	Benzidine
GC/MS	EPA 625 / 8270C,D	Benzo(a)anthracene
GC/MS	EPA 625 / 8270C,D	Benzo(a)pyrene
GC/MS	EPA 625 / 8270C,D	Benzo(b)fluoranthene
GC/MS	EPA 625 / 8270C,D	Benzo(g h i)perylene
GC/MS	EPA 625 / 8270C,D	Benzo(k)fluoranthene
GC/MS	EPA 8270C,D	Benzoic Acid
GC/MS	EPA 8270C,D	Benzyl alcohol
GC/MS	EPA 8270C,D	Biphenyl
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroethoxy)methane
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroethyl) ether
GC/MS	EPA 625 / 8270C,D	bis(2-Chloroisopropyl) ether (2, 2'-Oxybis(1-chloropropane))
GC/MS	EPA 625 / 8270C,D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625 / 8270C,D	Butyl benzyl phthalate
GC/MS	EPA 8270C,D	Carbazole
GC/MS	EPA 8270C,D	Chlorobenzilate
GC/MS	EPA 625 / 8270C,D	Chrysene
GC/MS	EPA 8270C,D	Diallate
GC/MS	EPA 625 / 8270C,D	Dibenz(a h)anthracene



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	Dibenzofuran
GC/MS	EPA 625 / 8270C,D	Diethyl phthalate
GC/MS	EPA 8270C,D	Dimethoate
GC/MS	EPA 625 / 8270C,D	Dimethyl phthalate
GC/MS	EPA 625 / 8270C,D	Di-n-butyl phthalate
GC/MS	EPA 625 / 8270C,D	Di-n-octyl phthalate
GC/MS	EPA 8270C,D	Ethyl methanesulfonate
GC/MS	EPA 8270C,D	Famfur
GC/MS	EPA 625 / 8270C,D	Fluoranthene
GC/MS	EPA 625 / 8270C,D	Fluorene
GC/MS	EPA 625 / 8270C,D	Hexachlorobenzene
GC/MS	EPA 625 / 8270C,D	Hexachlorobutadiene
GC/MS	EPA 625 / 8270C,D	Hexachlorocyclopentadiene
GC/MS	EPA 625 / 8270C,D	Hexachloroethane
GC/MS	EPA 8270C,D	Hexachloropropene
GC/MS	EPA 625 / 8270C,D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C,D	Isodrin
GC/MS	EPA 625 / 8270C,D	Isophorone
GC/MS	EPA 8270C,D	Isosafrole
GC/MS	EPA 8270C,D	Methapyriline
GC/MS	EPA 8270C,D	Methy methanesulfonate
GC/MS	EPA 8270C,D	Methyl parathion
GC/MS	EPA 625 / 8270C,D	Naphthalene
GC/MS	EPA 625 / 8270C,D	Nitrobenzene
GC/MS	EPA 8270C,D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C,D	n-Nitrosodiethylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodimethylamine
GC/MS	EPA 8270C,D	n-Nitroso-di-n-butylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodi-n-propylamine
GC/MS	EPA 625 / 8270C,D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C,D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C,D	n-Nitrosomorpholine
GC/MS	EPA 8270C,D	n-Nitrosopiperidine



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C,D	o o o-Triethyl phosphorothioate
GC/MS	EPA 8270C,D	o-Toluidine
GC/MS	EPA 8270C,D	Pentachlorobenzene
GC/MS	EPA 8270C,D	Pentachloronitrobenzene
GC/MS	EPA 625 / 8270C,D	Pentachlorophenol
GC/MS	EPA 8270C,D	Phenacetin
GC/MS	EPA 625 / 8270C,D	Phenanthrene
GC/MS	EPA 625 / 8270C,D	Phenol
GC/MS	EPA 8270C,D	Phorate
GC/MS	EPA 8270C,D	Pronamide
GC/MS	EPA 625 / 8270C,D	Pyrene
GC/MS	EPA 8270C,D	Pyrididine
GC/MS	EPA 8270C,D	Safrole
GC/MS	EPA 8270C,D	Thionazin
HPLC/UV	EPA 8330A/8330B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene
HPLC/UV	EPA 8330A/8330B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/8330B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/8330B	Tetryl

Non-Potable Water		
Technology	Method	Analyte
CVAA	EPA 245.1 / 7470A	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7 / 6010B,C	Aluminum
ICP/AES	EPA 200.7 / 6010B,C	Antimony
ICP/AES	EPA 200.7 / 6010B,C	Arsenic
ICP/AES	EPA 200.7 / 6010B,C	Barium
ICP/AES	EPA 200.7 / 6010B,C	Beryllium
ICP/AES	EPA 200.7 / 6010B,C	Boron
ICP/AES	EPA 200.7 / 6010B,C	Cadmium
ICP/AES	EPA 200.7 / 6010B,C	Calcium
ICP/AES	EPA 200.7 / 6010B,C	Chromium
ICP/AES	EPA 200.7 / 6010B,C	Cobalt
ICP/AES	EPA 200.7 / 6010B,C	Copper
ICP/AES	EPA 200.7 / 6010B,C	Iron
ICP/AES	EPA 200.7 / 6010B,C	Lead
ICP/AES	EPA 200.7 / 6010B,C	Magnesium
ICP/AES	EPA 200.7 / 6010B,C	Manganese
ICP/AES	EPA 200.7 / 6010B,C	Molybdenum
ICP/AES	EPA 200.7 / 6010B,C	Nickel
ICP/AES	EPA 200.7 / 6010B,C	Potassium
ICP/AES	EPA 200.7 / 6010B,C	Selenium
ICP/AES	EPA 200.7	Silicon
ICP/AES	EPA 200.7 / 6010B,C	Silver
ICP/AES	EPA 200.7 / 6010B,C	Sodium
ICP/AES	EPA 6010B,C	Strontium
ICP/AES	EPA 200.7 / 6010B,C	Thallium
ICP/AES	EPA 200.7 / 6010B,C	Tin
ICP/AES	EPA 200.7 / 6010B,C	Titanium
ICP/AES	EPA 200.7 / 6010B,C	Vanadium
ICP/AES	EPA 200.7 / 6010B,C	Zinc
ICP/MS	EPA 200.8 / 6020A	Aluminum
ICP/MS	EPA 200.8 / 6020A	Antimony
ICP/MS	EPA 200.8 / 6020A	Arsenic



Non-Potable Water		
Technology	Method	Analyte
ICP/MS	EPA 200.8 / 6020A	Barium
ICP/MS	EPA 200.8 / 6020A	Beryllium
ICP/MS	EPA 200.8 / 6020A	Boron
ICP/MS	EPA 200.8 / 6020A	Cadmium
ICP/MS	EPA 200.8 / 6020A	Calcium
ICP/MS	EPA 200.8 / 6020A	Chromium
ICP/MS	EPA 200.8 / 6020A	Cobalt
ICP/MS	EPA 200.8 / 6020A	Copper
ICP/MS	EPA 200.8 / 6020A	Iron
ICP/MS	EPA 200.8 / 6020A	Lead
ICP/MS	EPA 200.8 / 6020A	Magnesium
ICP/MS	EPA 200.8 / 6020A	Manganese
ICP/MS	EPA 200.8 / 6020A	Molybdenum
ICP/MS	EPA 200.8 / 6020A	Nickel
ICP/MS	EPA 200.8 / 6020A	Potassium
ICP/MS	EPA 200.8 / 6020A	Selenium
ICP/MS	EPA 200.8 / 6020A	Silicon
ICP/MS	EPA 200.8 / 6020A	Silver
ICP/MS	EPA 200.8 / 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 200.8 / 6020A	Thallium
ICP/MS	EPA 200.8 / 6020A	Tin
ICP/MS	EPA 200.8 / 6020A	Titanium
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8 / 6020A	Vanadium
ICP/MS	EPA 200.8 / 6020A	Zinc
IC	EPA 300.0 / 9056A	Bromide
IC	EPA 300.0 / 9056A	Chloride
IC	EPA 300.0 / 9056A	Nitrate as N
IC	EPA 300.0 / 9056A	Nitrite as N
IC	EPA 300.0 / 9056A	Nitrate + Nitrite
IC	EPA 300.0 / 9056A	Orthophosphate as P
IC	EPA 300.0 / 9056A	Sulfate



Non-Potable Water		
Technology	Method	Analyte
Titration	EPA 310.2 / SM 2320B	Alkalinity
Calculation	SM 2340C	Hardness
Gravimetric	EPA 1664A	Oil and Grease
Gravimetric	SM 2540B,C,D	Solids
ISE	EPA 120.1 / SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH
Titration	SM 2340B	Hardness
Titration	SM 4500SO ₃ B	Sulfite
Titration	EPA 9034 / SM 4500S ²⁻ E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
IR	EPA 9060A / SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1 / SM 2130B	Turbidity
Turbidimetric	EPA 9038 / ASTM 516-02	Sulfate
UV/VIS	EPA 335.4 / EPA 9012B / SM 4500-CN G	Amenable cyanide
UV/VIS	EPA 350.1 / SM 4500NH ₃ H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2 / SM 4500NO ₃ F	Nitrate + Nitrite
UV/VIS	EPA 353.2 / SM 4500NO ₃ F	Nitrate as N
UV/VIS	EPA 353.2 / SM 4500NO ₃ F	Nitrite as N
UV/VIS	EPA 365.1 / SM 4500P E	Orthophosphate as P
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 376.3	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1 / 9065	Total Phenolics
UV/VIS	SM 4500Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS



Non-Potable Water		
Technology	Method	Analyte
UV/VIS	EPA 7196A / SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B / 335.4	Total Cyanide
UV/VIS	EPA 9251 / SM 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	Method	Type
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	Method	Analyte
GC/ECD	EPA 8081B	4, 4'-DDD
GC/ECD	EPA 8081B	4, 4'-DDE
GC/ECD	EPA 8081B	4, 4'-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608 / 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin



Solid and Chemical Waste		
Technology	Method	Analyte
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4', 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)

Solid and Chemical Waste		
Technology	Method	Analyte
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C	Diesel range organics (DRO)
GC/FID	EPA 8015C	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics

Solid and Chemical Waste		
Technology	Method	Analyte
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B,C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B,C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B,C	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B,C	1, 1-Dichloroethane
GC/MS	EPA 8260B,C	1, 1-Dichloroethylene
GC/MS	EPA 8260B,C	1, 1-Dichloropropene
GC/MS	EPA 8260B,C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B,C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B,C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B,C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B,C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B,C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 2-Dichloroethane
GC/MS	EPA 8260B,C	1, 2-Dichloropropane
GC/MS	EPA 8260B,C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B,C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 3-Dichloropropane
GC/MS	EPA 8260B,C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B,C	1, 4-Dioxane
GC/MS	EPA 8260B,C	2, 2-Dichloropropane
GC/MS	EPA 8260B,C	2-Butanone
GC/MS	EPA 8260B,C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B,C	2-Chlorotoluene
GC/MS	EPA 8260B,C	2-Hexanone
GC/MS	EPA 8260B,C	4-Chlorotoluene
GC/MS	EPA 8260B,C	4-Methyl-2-pentanone
GC/MS	EPA 8260B,C	Acetone
GC/MS	EPA 8260B,C	Acetonitrile
GC/MS	EPA 8260B,C	Acrolein

Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8260B,C	Acrylonitrile
GC/MS	EPA 8260B,C	Allyl chloride
GC/MS	EPA 8260B,C	Benzene
GC/MS	EPA 8260B,C	Bromobenzene
GC/MS	EPA 8260B,C	Bromochloromethane
GC/MS	EPA 8260B,C	Bromodichloromethane
GC/MS	EPA 8260B,C	Bromoform
GC/MS	EPA 8260B,C	Carbon disulfide
GC/MS	EPA 8260B,C	Carbon tetrachloride
GC/MS	EPA 8260B,C	Chlorobenzene
GC/MS	EPA 8260B,C	Chloroethane
GC/MS	EPA 8260B,C	Chloroform
GC/MS	EPA 8260B,C	Chloroprene
GC/MS	EPA 8260B,C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B,C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B,C	Cyclohexane
GC/MS	EPA 8260B,C	Dibromochloromethane
GC/MS	EPA 8260B,C	Dibromomethane
GC/MS	EPA 8260B,C	Dichlorodifluoromethane
GC/MS	EPA 8260B,C	Diethyl ether
GC/MS	EPA 8260B,C	Di-isopropylether
GC/MS	EPA 8260B,C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B,C	Ethyl methacrylate
GC/MS	EPA 8260B,C	Ethylbenzene
GC/MS	EPA 8260B,C	Ethyl-t-butylether
GC/MS	EPA 8260B,C	Hexachlorobutadiene
GC/MS	EPA 8260B,C	Iodomethane
GC/MS	EPA 8260B,C	Isobutyl alcohol
GC/MS	EPA 8260B,C	Isopropyl benzene
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B,C	Methacrylonitrile
GC/MS	EPA 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B,C	Methyl chloride (Chloromethane)

Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8260B,C	Methyl methacrylate
GC/MS	EPA 8260B,C	Methyl tert-butyl ether
GC/MS	EPA 8260B,C	Methylcyclohexane
GC/MS	EPA 8260B,C	Methylene chloride
GC/MS	EPA 8260B,C	Naphthalene
GC/MS	EPA 8260B,C	n-Butylbenzene
GC/MS	EPA 8260B,C	n-propylbenzene
GC/MS	EPA 8260B,C	o-Xylene
GC/MS	EPA 8260B,C	p-Isopropyltoluene
GC/MS	EPA 8260B,C	Propionitrile
GC/MS	EPA 8260B,C	sec-butylbenzene
GC/MS	EPA 8260B,C	Styrene
GC/MS	EPA 8260B,C	t-Amylmethylether
GC/MS	EPA 8260B,C	tert-Butyl alcohol
GC/MS	EPA 8260B,C	tert-Butylbenzene
GC/MS	EPA 8260B,C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B,C	Tetrahydrofuran
GC/MS	EPA 8260B,C	Toluene
GC/MS	EPA 8260B,C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B,C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B,C	Trans-1, 4-Dichloro-2-butene
GC/MS	EPA 8260B,C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B,C	Trichlorofluoromethane
GC/MS	EPA 8260B,C	Vinyl acetate
GC/MS	EPA 8260B,C	Vinyl chloride
GC/MS	EPA 8260B,C	Xylene
GC/MS	EPA 8270C,D	1-Naphthylamine
GC/MS	EPA 8270C,D	2-Acetylaminofluorene
GC/MS	EPA 8270C,D	2-Chloronaphthalene
GC/MS	EPA 8270C,D	2-Chlorophenol
GC/MS	EPA 8270C,D	2-Methylnaphthalene
GC/MS	EPA 8270C,D	2-Methylphenol
GC/MS	EPA 8270C,D	2-Naphthylamine

Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	2-Nitroaniline
GC/MS	EPA 8270C,D	2-Nitrophenol
GC/MS	EPA 8270C,D	2-Picoline
GC/MS	EPA 8270C,D	3-Methylcholanthrene
GC/MS	EPA 8270C,D	3-Nitroaniline
GC/MS	EPA 8270C,D	4-Aminobiphenyl
GC/MS	EPA 8270C,D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C,D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C,D	4-Chloroaniline
GC/MS	EPA 8270C,D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C,D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C,D	4-Methylphenol
GC/MS	EPA 8270C,D	4-Nitroaniline
GC/MS	EPA 8270C,D	4-Nitrophenol
GC/MS	EPA 8270C,D	5-Nitro-o-toluidine
GC/MS	EPA 8270C,D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C,D	Acenaphthene
GC/MS	EPA 8270C,D	Acenaphthylene
GC/MS	EPA 8270C,D	Acetophenone
GC/MS	EPA 8270C,D	Aniline
GC/MS	EPA 8270C,D	Anthracene
GC/MS	EPA 8270C,D	Aramite
GC/MS	EPA 8270C,D	Atrazine
GC/MS	EPA 8270C,D	Benzidine
GC/MS	EPA 8270C,D	Benzo(a)anthracene
GC/MS	EPA 8270C,D	Benzo(a)pyrene
GC/MS	EPA 8270C,D	Benzo(b)fluoranthene
GC/MS	EPA 8270C,D	Benzo(g h i)perylene
GC/MS	EPA 8270C,D	Benzo(k)fluoranthene
GC/MS	EPA 8270C,D	Benzoic Acid
GC/MS	EPA 8270C,D	Benzyl alcohol
GC/MS	EPA 8270C,D	Biphenyl
GC/MS	EPA 8270C,D	bis(2-Chloroethoxy)methane

Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C,D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270C,D	Butyl benzyl phthalate
GC/MS	EPA 8270C,D	Carbazole
GC/MS	EPA 8270C,D	Chlorobenzilate
GC/MS	EPA 8270C,D	Chrysene
GC/MS	EPA 8270C,D	Diallate
GC/MS	EPA 8270C,D	Dibenz(a h)anthracene
GC/MS	EPA 8270C,D	Dibenzofuran
GC/MS	EPA 8270C,D	Diethyl phthalate
GC/MS	EPA 8270C,D	Dimethoate
GC/MS	EPA 8270C,D	Dimethyl phthalate
GC/MS	EPA 8270C,D	Di-n-butyl phthalate
GC/MS	EPA 8270C,D	Di-n-octyl phthalate
GC/MS	EPA 8270C,D	Ethyl methanesulfonate
GC/MS	EPA 8270C,D	Famfur
GC/MS	EPA 8270C,D	Fluoranthene
GC/MS	EPA 8270C,D	Fluorene
GC/MS	EPA 8270C,D	Hexachlorobenzene
GC/MS	EPA 8270C,D	Hexachlorobutadiene
GC/MS	EPA 8270C,D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C,D	Hexachloroethane
GC/MS	EPA 8270C,D	Hexachloropropene
GC/MS	EPA 8270C,D	Isodrin
GC/MS	EPA 8270C,D	Isophorone
GC/MS	EPA 8270C,D	Isosafrole
GC/MS	EPA 8270C,D	Methapyriline
GC/MS	EPA 8270C,D	Methyl methanesulfonate
GC/MS	EPA 8270C,D	Methyl parathion
GC/MS	EPA 8270C,D	Naphthalene
GC/MS	EPA 8270C,D	Nitrobenzene
GC/MS	EPA 8270C,D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C,D	n-Nitrosodiethylamine

Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	n-Nitrosodimethylamine
GC/MS	EPA 8270C,D	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270C,D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C,D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C,D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C,D	n-Nitrosomorpholine
GC/MS	EPA 8270C,D	n-Nitrosopiperidine
GC/MS	EPA 8270C,D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C,D	o o o-Triethyl phosphorothioate
GC/MS	EPA 8270C,D	o-Toluidine
GC/MS	EPA 8270C,D	Pentachlorobenzene
GC/MS	EPA 8270C,D	Pentachloronitrobenzene
GC/MS	EPA 8270C,D	Pentachlorophenol
GC/MS	EPA 8270C,D	Phenacetin
GC/MS	EPA 8270C,D	Phenanthrene
GC/MS	EPA 8270C,D	Phenol
GC/MS	EPA 8270C,D	Phorate
GC/MS	EPA 8270C,D	Pronamide
GC/MS	EPA 8270C,D	Pyrene
GC/MS	EPA 8270C,D	Pyrididne
GC/MS	EPA 8270C,D	Safrole
GC/MS	EPA 8270C,D	Thionazin
GC/MS	EPA 8270C,D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C,D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C,D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C,D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C,D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C,D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C,D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C,D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C,D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 3-Dinitrobenzene



Solid and Chemical Waste		
Technology	Method	Analyte
GC/MS	EPA 8270C,D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C,D	1, 4-Dioxane
GC/MS	EPA 8270C,D	1, 4-Naphthoquinone
GC/MS	EPA 8270C,D	1, 4-Phenylenediamine
GC/MS	EPA 8270C,D	bis(2-Chloroisopropyl) ether (2, 2'-Oxybis(1-chloropropane))
GC/MS	EPA 8270C,D	2, 4-Dichlorophenol
GC/MS	EPA 8270C,D	2, 4-Dimethylphenol
GC/MS	EPA 8270C,D	2, 4-Dinitrophenol
GC/MS	EPA 8270C,D	2, 4-Dinitrotoluene (2 4-DNT)
GC/MS	EPA 8270C,D	2, 6-Dichlorophenol
GC/MS	EPA 8270C,D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C,D	3, 3'-Dichlorobenzidine
GC/MS	EPA 8270C,D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C,D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C,D	7,12-Dimethylphenethylamine
HPLC/UV	EPA 8330A	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	4-Amino-2,3-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl



Solid and Chemical Waste		
Technology	Method	Analyte
HPLC/UV	8330B (W/O Soil Grinding)	1, 3, 5-Trinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	1, 3-Dinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4, 6-Trinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 6-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Amino-4, 6 -dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Amino-2,3-dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	Ethylene glycol dinitrate (EGDN)
HPLC/UV	8330B (W/O Soil Grinding)	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	8330B (W/O Soil Grinding)	Nitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	Nitroglycerin
HPLC/UV	8330B (W/O Soil Grinding)	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	8330B (W/O Soil Grinding)	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	8330B (W/O Soil Grinding)	Tetryl
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010B,C	Aluminum
ICP/AES	EPA 6010B,C	Antimony
ICP/AES	EPA 6010B,C	Arsenic
ICP/AES	EPA 6010B,C	Barium
ICP/AES	EPA 6010B,C	Beryllium
ICP/AES	EPA 6010B,C	Boron
ICP/AES	EPA 6010B,C	Cadmium
ICP/AES	EPA 6010B,C	Calcium
ICP/AES	EPA 6010B,C	Chromium
ICP/AES	EPA 6010B,C	Cobalt
ICP/AES	EPA 6010B,C	Copper
ICP/AES	EPA 6010B,C	Iron
ICP/AES	EPA 6010B,C	Lead
ICP/AES	EPA 6010B,C	Magnesium



Solid and Chemical Waste		
Technology	Method	Analyte
ICP/AES	EPA 6010B,C	Manganese
ICP/AES	EPA 6010B,C	Molybdenum
ICP/AES	EPA 6010B,C	Nickel
ICP/AES	EPA 6010B,C	Potassium
ICP/AES	EPA 6010B,C	Selenium
ICP/AES	EPA 200.7	Silicon
ICP/AES	EPA 6010B,C	Silver
ICP/AES	EPA 6010B,C	Sodium
ICP/AES	EPA 6010B,C	Strontium
ICP/AES	EPA 6010B,C	Thallium
ICP/AES	EPA 6010B,C	Tin
ICP/AES	EPA 6010B,C	Titanium
ICP/AES	EPA 6010B,C	Vanadium
ICP/AES	EPA 6010B,C	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium

Solid and Chemical Waste		
Technology	Method	Analyte
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9070A / 9071B	Oil and Grease
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038 / ASTM 516-02	Sulfate
UV/VIS	EPA 350.1 / SM 4500NH3 H	Ammonia as N
UV/VIS	EPA 9251 / SM 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	EPA 376.3	AVS-SEM
UV/VIS	SM 3500Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196	Chromium VI
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Preparation	Method	Type
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up
Cleanup Methods	EPA 3620C	Florsil Clean-up

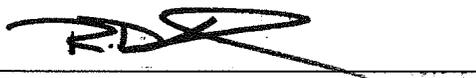


Solid and Chemical Waste		
Technology	Method	Analyte
Preparation	Method	Type
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: _____


R. Douglas Leonard
Chief Technical Officer

Date: January 20, 2011

Issued: 11/04/09
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Revised: 04/06/10

Revised: 9/9/10

Revised: 10/13/10

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 2/15/01

Operations Manager: John C. Burton Date: 2/14/01

QA Officer: Deborah J. Madreau Date: 2.14.01

General Manager: Deborah J. Madreau Date: 2/14/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, other updated changes to all sections.	DN	2.14.01	2/14/01
02	Minor changes to sections 5, 6 + 7. Grammatical errors corrected	DN	4.9.02	4.9.02
03	added definitions modified Sections 5, 7, 8 and 9 modified table to include extra compounds	LAD	01-31-05	01-31-05
04	Added pollution control and waste disposal to Sect. 1A. Added kims definition to sect. 1.1 and 7.9.1. Removed all references to 60ml vials. Edited AQ LCS prep. minor changes throughout to reflect current practice. Fixed numbering and formatting	LAD	09/07	09/07
05	Corrected references in text to Tables 1, 3 & 4. Reworded Sects 8.14.4, 8.1.5, 8.4.2.3 and 8.4.3 for clarity. Changed lab fortified blank to LCS and lab fortified matrix to MS	LAD	03/08	03/08

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (MADEP - VPH)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

1.0 SCOPE AND APPLICATION

This method is designed to measure the collective concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. Volatile aliphatic hydrocarbons are collectively quantitated within two ranges: C₅ through C₈, and C₉ through C₁₂. Volatile aromatic hydrocarbons are collectively quantitated within the C₉ to C₁₀ range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 36°C and 220°C.

This method is based on a purge-and-trap, gas chromatography (GC) procedure using in-series Photoionization and Flame Ionization Detectors (PID/FID). This method should be used by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

This method is also able to measure the individual concentrations of the VPH Target Analytes benzene, toluene, ethylbenzene, xylenes (BTEX), naphthalene, and methyl-tert-butylether (MTBE) in water and soil. Use of this method to identify and quantitate these Target Analytes is optional.

Petroleum products suitable for evaluation by this method include gasoline, mineral spirits, and certain petroleum naphthas. This method, in and of itself, is not suitable for the evaluation of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products that contain a significant percentage of hydrocarbons heavier than C₁₂.

This method includes a series of data manipulation steps to determine the concentrations of aliphatic and aromatic ranges of interest.

Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target Analyte. Confirmatory analysis by a GC/MS procedure or other suitable method is recommended in cases where a Target Analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where co-elution of a non-targeted hydrocarbon compound is suspected.

This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance. MADEP encourages methodological innovations which (a) better achieve method and/or data quality objectives, (b) increase analytical precision and accuracy, (c) reduce analytical uncertainties and expenses, and/or (d) reduce the use of toxic solvents and generation of hazardous wastes. Laboratories that modify this method must achieve all required performance and acceptance standards, and must have on file a Standard Operating Procedure which thoroughly describes the revised

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

or alternative method, and documentation which demonstrates an equivalent or superior level of performance. All significant modifications to the method must be disclosed and described on the data report form.

1.1 Definitions

Analytical Batch is defined as a group of field samples with similar matrices which are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

Calibration Check Standard is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

Calibration Standards are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

C₅ through C₈ Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram from n-pentane (C₅) to just before n-nonane (C₉).

C₉ through C₁₂ Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram from n-nonane (C₉) to just before naphthalene.

C₉ through C₁₀ Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds which elute on the PID chromatogram from just after o-xylene to just before naphthalene. Although it is an aromatic compound with 10 carbon atoms, naphthalene is excluded from this range because it is evaluated as a separate (Target) analyte.

Field Duplicates are defined as two separate samples collected at the same time and place under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

Laboratory Control Sample (LCS) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution.

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

Laboratory Duplicates are defined as split samples taken from the same sampling container and analyzed separately with identical procedures. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

Matrix Spike (MS) Sample is defined as an environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The MS sample is treated and analyzed exactly as a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the MS sample corrected for background concentrations.

Laboratory Method Blank (LMB) is defined as an aliquot of laboratory reagent grade water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

Matrix Spiking Solution is defined as a solution prepared independently from the calibration standards, containing known concentrations of method analytes.

System Solvent Blank is defined as an aliquot of method solvent (e.g., methanol) that is directly purged into the GC system. The purpose of the Solvent Blank is to determine the level of noise and baseline rise attributable solely to the GC system, in the absence of any other analytes or contaminants.

Target VPH Analytes are defined as benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, naphthalene, and methyl-tert-butylether.

Unadjusted C₅ through C₈ Aliphatic Hydrocarbons are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-pentane (C₅) to just before n-nonane (C₉).

Unadjusted C₉ through C₁₂ Aliphatic Hydrocarbons are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-nonane (C₉) to just before naphthalene.

Volatile Petroleum Hydrocarbons (VPH) are defined as collective fractions of hydrocarbon compounds eluting from n-pentane to naphthalene, excluding Target VPH Analytes. VPH is comprised of C₅ through C₈ Aliphatic Hydrocarbons, C₉ through C₁₂ Aliphatic Hydrocarbons, and C₉ through C₁₀ Aromatic Hydrocarbons.

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Volatile Petroleum Hydrocarbon (VPH) Component Standard is defined as a 15 component mixture of the aliphatic and aromatic compounds listed in Table 3. The compounds comprising the VPH Component Standard are used to (a) define the individual retention times and chromatographic response factors for each of the Target VPH Analytes, (b) define and establish the windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and (c) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing laboratory status reports, and generating final reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of VPH by MADEP VPH-04-1.1. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of VPH by MADEP VPH-04-1.1 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

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It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP. Purge vial and methanol waste are disposed of in the "A" waste satellite accumulation area located between GC04 and GC09.

2.0 SUMMARY OF METHOD

- 2.1 Samples are analyzed using purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic

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compounds. Detection is achieved by a Photoionization detector (PID) and flame ionization detector (FID) in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations of targeted analytes and collective concentration of aromatic hydrocarbons within the C₉ through C₁₀ range. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C₅ through C₈ and C₉ through C₁₂ ranges.

- 2.2 This method is suitable for the analysis of waters, soils, and sediments. Water samples may be analyzed directly for volatile petroleum hydrocarbons by purge-and-trap concentration and gas chromatography. Soil samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is then analyzed by purge-and-trap GC.
- 2.3 This method is based on (1) USEPA Methods 5030, 8000, 8020, and 8015, SW-846, "Test Methods for Evaluating Solid Wastes", 3rd Edition, 1986; (2) Draft "Method for Determination of Gasoline Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Gasoline Range Organics", Wisconsin Department of Natural Resources, PUBL-SW140, 1992.

3.0 INTERFERENCES

- 3.1 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage or by dissolution of volatiles into the methanol used for preservation. Trip blanks prepared from both laboratory reagent grade water and methanol should be carried through sampling and subsequent storage and handling to serve as a check on such contamination.
- 3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with laboratory reagent grade water or solvent. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling-point compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in an oven at 105°C between analyses. The trap and other parts of the system are also subject to contamination, therefore, frequent bake-out and purging of the entire system may be required. A screening step is recommended to protect analytical instrumentation. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of a system solvent blank or laboratory method blank to check for cross-contamination.

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- 3.3 Certain organic compounds not associated with releases of petroleum products, including chlorinated solvents, ketones, and ethers, will be quantitated as Volatile Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.
- 3.4 The response selectivity of a photoionization detector (PID) is used in this method to differentiate aromatic hydrocarbons from aliphatic hydrocarbons. All compounds eluting on the PID chromatogram after o-xylene are identified by the method as aromatic hydrocarbons. This will lead to an overestimation of aromatic hydrocarbons within samples, as certain aliphatic compounds will elicit a response on the PID, particularly unsaturated compounds such as alkenes. The significance and implications of this overestimation will vary from sample to sample; where less conservative data are desired, additional actions should be considered to minimize the detection of non-aromatic compounds, including the use of a lower energy PID lamp, different chromatographic columns, and/or addition of a pre-analysis sample cleanup step.
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4.0 APPARATUS AND MATERIALS

- 4.1 The following glassware is used in this method:
- 4.1.1 VOC Vials: 40 mL VOC vials with Teflon/ silicone septa for waters and soils.
 - 4.1.2 Class "A" Volumetric flasks: 10 mL, 50 mL, 100 mL, and 1,000 mL with a ground-glass stopper.
 - 4.1.3 Disposable pipettes: Pasteur.
- 4.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples.
- 4.3 Gas Chromatography
- 4.3.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph and purge-and-trap concentrator. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

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- 4.3.2 Chromatographic Column: The required column is: 105M x 0.53 mm I.D. Restek RTX 502.2 with 3 micron film thickness, or column with equivalent chromatographic properties.

NOTE: Based upon data obtained from the Round Robin testing programs, the choice of chromatographic column may have a significant impact on the apportionment and quantitation of aliphatic and aromatic compounds within the fractional ranges specified in this method. Substitution of the required column is not allowed, unless it can be demonstrated that the selected column has equivalent chromatographic properties and retention times for the aliphatic and aromatic compounds and ranges of interest. To demonstrate equivalency of column chromatography, a neat gasoline standard must be analyzed on both the required column and the proposed substitute column, with all other run and system parameters held constant. The concentrations of C₅-C₈ and C₉-C₁₂ Aliphatic Hydrocarbons must be determined for each column (in which the concentration of the Target/aromatic analytes have been subtracted from the GC/FID response). The Relative Percent Difference between the concentrations of each fraction obtained for each column must be equal to or less than 25%.

- 4.3.3 Detectors: The method requires the use of a Photoionization Detector (PID) in series with a Flame Ionization Detector (FID); the PID first in the series. The method is based upon the use of a 10.0 +/- eV PID lamp, although lower energy lamps are permissible in order to minimize PID response to aliphatic compounds. In lieu of an in-series arrangement, in-parallel PID and FID units may be also used.
- 4.3.4 Purge-and-trap device: The purge-and-trap device consists of a sample purger, a trap, and a desorber. Several complete devices are commercially available.

4.3.4.1 The purging chamber must be designed to accept 5 mL samples with a water column at least 3 cm deep. Purging devices larger than 5 mL have a reduced purging efficiency and should not be used. The gaseous headspace between the water column and the top of the vessel should be at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. Fritted glass or needle sparge cells may be used. If needle sparge cells are used, the purge gas must be introduced no more than 5 mm from the base of the water column. Alternate sample

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purge devices may be used, provided equivalent performance is demonstrated.

4.3.4.2 The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inches. The trap should be packed with 400 mg of Carbopack B (Supelco Cat. No. 209273). Alternative trap packing materials include: Tenax GC (or equivalent); 7.6 cm Carbopack B and 1.3 cm Carbosieve S-III (Supelco Cat No. 2-0321); 7 cm Carbopack C and 1.2 cm Carbopack B (Supelco Cat No. 2-1064); or equal volumes of Tenax, silica gel, and charcoal as described in EPA SW-846 Method 5030. In general, Carbopack trap packing materials are recommended because they have less of a tendency to retain methanol, which could interfere with the elution of pentane and quench the FID flame. The trap length and packing materials may be varied as long as equivalent performance has been verified.

4.3.4.3 Prior to initial use, the Carbopack B trap should be conditioned overnight at 270°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to a hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min. at 260°C with backflushing. The trap may be vented to the analytical column during daily conditioning, however, the column must be run through the temperature program prior to analysis of samples. Devices other than the traps recommended in Section 4.3.4.2 should be conditioned and desorbed according to the manufacturer's guidelines.

4.3.4.4 The desorber should be capable of rapidly heating the trap to 240°C for desorption.

- 4.4 Data System: A data system which allows the continuous acquisition of data throughout the duration of the chromatographic program must be interfaced to the GC. The data system must be capable of storing and re-integrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. All data editing will be reviewed by the Department Manager or qualified designee before samples are reported.
- 4.5 Ultrasonic bath.
- 4.6 Syringes: 5 mL Luerlock glass hypodermic and 5 mL gas-tight syringe with shutoff valve.
- 4.7 Syringe valve: Two-way, with luer ends.
- 4.8 Microsyringes: 1 µL, 5 µL, 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, and 1,000 µL.

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4.9 Spatula: Stainless steel.

5.0 REAGENTS

5.1 Reagents

- 5.1.1 Organic-free water: carbon filtered ASTM Type II water (laboratory reagent grade water)
- 5.1.2 Methanol: purge-and-trap grade or equivalent; store away from other solvents to prevent cross-contamination
- 5.1.3 Organic-free sand: sand baked in an oven at 400°C for four hours to remove organic contaminants

5.2 Stock Standard Solution

- 5.2.1 The stock standard solution consists of the 15 VPH component standards and a surrogate standard that is purchased from vendors like ULTRA Scientific at a concentration of 1000 µg/mL. The solution is stored at -10°C to -20°C and protected from light.
- 5.2.2 The stock standard solution must be replaced after six months from the date of opening or sooner if the manufacture's date is less.

5.3 Primary Dilution Standard

- 5.3.1 The primary dilution standards are prepared in methanol at concentrations listed in Table 3. These standards are stored at -10°C to -20°C.
- 5.3.2 The primary dilution standards should be replaced at least monthly.

5.4 VPH Calibration Standards

VPH calibration standards are prepared by injecting an aliquot of either the primary dilution standard or the stock standard directly into the 5 mL syringe. The volumes added are listed in Table 3.

5.5 Surrogate Standard

- 5.5.1 The analyst must monitor both the performance of the analytical system and the effectiveness of the method in dealing with sample matrices by spiking each sample, blank, and matrix spike with a surrogate standard. The

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surrogate standard is also added to the VPH calibration standard solutions. The recommended surrogate standard is 2,5-dibromotoluene, which elutes after all aliphatic and aromatic compounds of interest. The use of another surrogate compound is permissible.

5.5.2 Surrogate Spiking Solution

5.5.2.1 For aqueous samples, from a stock standard solution prepare a surrogate spiking solution at 50 µg/mL in methanol. Add 5 µL of this surrogate spiking solution directly into the 5 mL syringe with every aqueous sample, method blank, and matrix spike. This will correlate to a concentration of 50 µg/L in the sample.

5.5.2.2 For soil samples, using a 200 µg/mL stock solution add 1 mL to every soil sample, method blank, and matrix spike during the extraction step. This will correlate to a concentration of 50 µg/L in the sample for a 15 g extraction.

5.6 Matrix Spiking Solution

5.6.1 The matrix spiking solution consists of the Targeted VPH analytes.

5.6.1.1 For aqueous samples, using a stock standard solution at a concentration of 50 µg/mL in methanol, add 10 µL directly into the 5 mL syringe with every aqueous laboratory control sample (LCS) and matrix spike sample (MS). This will correlate to a concentration of 100 µg/L in the sample.

5.6.1.2 For soil samples, using a stock solution at a concentration of 500/1000/1500 µg/mL in methanol, add 500 µL to every LCS and MS.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous Samples

6.1.1 Aqueous samples should be collected in duplicate (or the number of vials directed by the laboratory) without agitation and without headspace in contaminant-free glass VOC vials with Teflon-lined septa screw caps. The Teflon liner must contact the sample. Samples must be acidified to a pH of 2 or less at the time of collection. This can generally be accomplished by adding 3 or 4 drops (0.1 to 0.2 mL) of 1:1 HCl (1 part laboratory reagent

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grade water and 1 part concentrated HCl) to a 40 mL sample vial. Samples must be cooled to 4°C immediately after collection.

- 6.1.2 A chain of custody form must accompany all sampling vials and must document the date and time of sample collection and acid preservation. The pH of all water samples must be determined by the laboratory unless sample vials containing acid for field preservation were supplied by the laboratory (this must be noted on the chain of custody). The pH measurement may be performed on left over sample. Any sample found to contain a pH above 2 must be so noted on the laboratory/data report sheet.
- 6.1.3 A laboratory reagent grade water trip blank should accompany each batch of water samples.
- 6.1.4 Any sample received by the laboratory that is not packed in ice or cooled to 4°C must be so noted on the laboratory/data report sheet.
- 6.1.5 Aqueous samples must be analyzed within 14 days of collection.

6.2 Soil Samples

- 6.2.1 Soil samples must be collected in a manner that minimizes sample handling and agitation. The use of specially designed air-tight collection samplers or a 30 mL plastic syringe with the end sliced off is recommended. All sediment must be removed from the glass threads of the vial to ensure an adequate seal. Samples must be cooled to 4°C immediately after collection.
- 6.2.2 **Methanol preservation of soil samples is mandatory.** Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air-tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection. Additional details and recommendations on soil sampling may be found in method MADEP VPH-04-1.1.
- 6.2.3 The desired ratio of methanol-to-soil is 1 mL methanol/1 gram soil, \pm 25%. The exact weight of the soil sample and volume of methanol must be known or ascertained by the laboratory when calculating and reporting soil concentration data. A recommended practice is for a laboratory to provide to a field sampling technician labeled, pre-weighed sampling vials with a measured volume of methanol, and a scribed mark indicating the level of methanol that should exist in the vial when the required quantity of soil sample has been added. This requires an estimate on the density and

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moisture content of the soil sample; a good estimate for most soils is 8-10 mL of displaced volume for 15 grams soil. **In all cases, the soil sample in the vial must be completely covered by methanol.**

- 6.2.4 Samples for VPH analysis should be collected in duplicate 40 mL vials. An additional sample of the soil must also be obtained (without methanol) to allow for a determination of soil moisture content and VPH dry weight correction factors.
- 6.2.5 A methanol trip blank should accompany each batch of soil samples.
- 6.2.6 A chain of custody form must accompany all sampling vials and must document the date and time of sample collection and, where appropriate, the volume of methanol added. Observations of vial leakage must be so noted on the laboratory/data report sheet.
- 6.2.7 Soil samples must be analyzed within 28 days of collection.
- 6.3 A summary of sample collection, preservation and holding times is provided in Table 4.

7.0 PROCEDURES

- 7.1 Sample Preparation and Purging
 - 7.1.1 It is highly recommended that all samples be screened prior to analysis. This screening step may be analysis of a soil sample's methanol extract (diluted) or the hexadecane extraction and screening method (SW-846 Method 3820).
 - 7.1.2 Water Samples - Introduce volatile compounds into the gas chromatograph using a purge-and-trap concentrator.
 - 7.1.2.1 Remove the plunger from a 5 mL syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample and vent any residual air while adjusting the sample volume to 5 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if a second analysis is needed an alternate vial will be used. If an alternate vial is not available then the compromised vial will be used and notated.

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- 7.1.2.2 If necessary, samples should be diluted prior to injection into the purge chamber. In such cases, all steps must be performed without delay until the diluted sample is in a gas-tight syringe.
- 7.1.2.2.1 Dilutions may be made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for highly concentrated samples.
- 7.1.2.2.2 Calculate the approximate volume of laboratory reagent grade water to be added to the volumetric flask selected and add slightly less than this volume of laboratory reagent grade water to the flask.
- 7.1.2.2.3 Inject the proper aliquot of sample from the syringe prepared in Paragraph 7.1.2.1 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with laboratory reagent grade water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions. Alternatively the dilutions can be made directly in the glass syringe to avoid further loss of volatiles.
- 7.1.2.2.4 Fill a 5 mL syringe with diluted sample as in Paragraph 7.1.2.1.
- 7.1.2.3 Add 5 μ L of the surrogate spiking solution through the valve bore of the syringe.
- 7.1.2.4 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.
- 7.1.2.5 Close the valve and start the LSC 3000 sample concentrator. Refer to the instrument maintenance logbook for the current purge-and-trap operating parameters.
- 7.1.2.6 If the concentration of an analyte or hydrocarbon fraction in a sample exceeds the calibration range, a dilution of the sample is required. If a sample analysis results in a saturated detector response for a compound, the analysis must be followed by a blank laboratory reagent grade water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

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7.1.2.7 All dilutions should keep the detector response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.1.3 Soil/Sediments - Soil and sediment samples are extracted with methanol. An aliquot of the extract is added to laboratory reagent grade water and introduced into the gas chromatograph using a purge-and-trap concentrator.

7.1.3.1 Weigh the sample vial to 0.1 g in a top loading balance and determine the weight of the soil/sediment sample; this determination requires knowledge of the empty/tarred weight of the sample vial and volume/weight of methanol preservative that was added to the sample vial.

7.1.3.2 Add 1 mL of the surrogate spiking solution. The concentration and/or volume of the surrogate spiking compound may need to be increased for samples that are highly contaminated (based upon screening and/or visual/olfactory evidence), to prevent dilution to below detectable limits. Shake the sample for 2 minutes and sonicate for 20 minutes.

7.1.3.3 Allow sediment to settle until a layer of methanol is apparent.

7.1.3.4 Using a microliter syringe, withdraw a 20 uL aliquot of the methanol extract for sparging. Sample screening data can be used to determine the volume of methanol extract to add to the 5 mL of laboratory reagent grade water for analysis. All dilutions must keep the response of the major constituents in the upper half of the linear range of the calibration curve.

7.1.3.5 Remove the plunger from one 5 mL Luerlock type syringe and fill until overflowing with laboratory reagent grade water. Replace the plunger and compress the water to vent trapped air. Pull the plunger to 5 mL for addition of the sample extract. Add the volume of methanol extract (20 µL maximum).

7.1.3.6 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.

7.1.4 Proceed with the analysis as described in Sections 7.1.2.5 through 7.1.2.7. Analyze all laboratory method blanks and QC samples under the same conditions as that used for samples.

7.1.5 If the responses exceed the calibration or linear range of the system, use a smaller aliquot of methanol.

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7.2 GC Conditions

7.2.1 Refer to instrument maintenance logbook for the current oven conditions. The typical oven temperature program is: Oven temperature 35°C, hold for 8 min, then to 90°C at 3°C/min, to 140°C at 5°C/min, to 230°C at 45°C/min; hold for 10.5 min. Conditions may be altered to improve resolution of volatile petroleum hydrocarbons.

7.2.2 Gas Flows: The recommended carrier gas is helium.

7.2.2.1 Carrier gas flow: 10 mL/min.

7.2.2.2 Air: 160 mL/min

7.2.2.3 Hydrogen: 30 mL/min

7.2.2.4 Make up gas flow: 15 mL/min

7.2.3 Miscellaneous:

7.2.3.1 FID temperature: 220°C

7.2.3.2 PID temperature: 220°C

7.2.3.3 Injection port temperature: 200°C

7.2.3.4 Column head pressure: 30 psi

7.3 Retention Time Windows

7.3.1 Before establishing retention time windows, make sure the GC system is within optimum operating conditions. Make three injections of the VPH Component Standard throughout the course of a 72 hr period. Serial injections over less than a 72 hr period may result in retention time windows that are too tight.

7.3.2 Calculate the standard deviation of the three absolute retention times for each individual compound in the VPH Component Standard.

7.3.3 The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each standard. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

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- 7.3.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.
- 7.3.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the lab.
- 7.3.6 VPH retention time (Rt) windows of the aliphatic ranges are defined as beginning 0.1 minutes before the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound, except for C₉, which is both a beginning and ending marker compound for two different ranges.

The C₅-C₈ Aliphatic Hydrocarbon range ends immediately (0.1 min) before the elution of the n-C₉ peak. The C₉-C₁₂ Aliphatic Hydrocarbon range begins 0.1 min before the elution of n-C₉, therefore there is no overlap of the two ranges and the n-C₉ peak is only included in the C₉-C₁₂ Aliphatic Hydrocarbon range.

The VPH retention time (Rt) window for the C₉-C₁₀ Aromatic Hydrocarbons is defined as beginning 0.1 minutes after the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound, VPH marker compounds and windows are summarized below.

VPH Marker Compounds

Hydrocarbon Range	Beginning Marker Compound	Ending Marker Compound
C ₅ - C ₈ Aliphatic Hydrocarbons (FID)	0.1 min before Pentane	0.1 min before n-Nonane
C ₉ - C ₁₂ Aliphatic Hydrocarbons (FID)	0.1 min before n-Nonane	0.1 min before Naphthalene ¹
C ₉ -C ₁₀ Aromatic Hydrocarbons (PID)	0.1 min after o-xylene	0.1 min before Naphthalene ¹

¹The retention time for Dodecane (C₁₂) is approximately 2 minutes less than the retention time for naphthalene, using the column and chromatographic conditions recommended for this method. For simplicity, naphthalene is used as the ending marker for the C₉-C₁₂ Aliphatic Hydrocarbon range.

7.4 External Standard Calibration Procedure

- 7.4.1 Analyze each of the six VPH Calibration standards following the procedure outlined in section 7.5. Tabulate the area responses against the concentration injected. The ratio of area response to the concentration

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injected, defined as the calibration factor (CF), may be calculated for target VPH analytes using the equation below.

Calibration Factor (CF) = area of peak/concentration purged ($\mu\text{g/L}$)

The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest, as determined using the equation below. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.

$\%RSD = (\text{stand dev of 6 CFs}/\text{mean of 6 CFs}) \times 100$

- 7.4.2 A collective calibration factor must also be established for each hydrocarbon range of interest: C₅-C₈ Aliphatic Hydrocarbons, C₉-C₁₂ Aliphatic Hydrocarbons and the C₉-C₁₀ Aromatic Hydrocarbons. Calculate the collective CFs for C₅-C₈ Aliphatic Hydrocarbons and C₉-C₁₂ Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CFs for the C₉-C₁₀ Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The results can be used to calculate the ratio of the peak area response summation to the concentration injected, defined as the CF, for the hydrocarbon ranges using the equation below. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest, as determined using the equation in 7.4.1.1.

CF = area summation of range components/total conc. purged ($\mu\text{g/L}$)

- 7.4.3 At a minimum, the working calibration curve or calibration factor must be verified on each working day, after every 20 samples and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than $\pm 25\%$, as determined using the equation below, a new calibration curve must be prepared for that analyte. Greater differences are permissible for n-nonane ($\pm 30\%$).

$\%D = (CF_{AVG} - CF_{CC}) / ((CF_{AVG}) \times 100)$

where:

CF_{AVG} = Average calibration factor calculated from initial calibration.
CF_{CC} = Calibration factor calculated from continuing calibration.

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- 7.4.4 Target VPH Analytes and C₉ to C₁₀ Aromatic Hydrocarbons are quantitated on the PID chromatogram.
- 7.4.5 C₅ through C₈ and C₉ through C₁₂ Aliphatic Hydrocarbons are quantitated on the FID chromatogram.
- 7.4.6 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, and surrogates at mid-calibration range concentration and is obtained from a source independent of the initial calibration source. Please refer to Table 1 for acceptance criteria and corrective action for this standard. When analyzed after a initial calibration and directly before samples, this standard also doubles as an LCS sample.

7.5 GC Analysis

- 7.5.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by samples interspersed with blanks and QC samples. The sequence ends when the set of samples has been analyzed or when qualitative and/or quantitative QC criteria are exceeded.
- 7.5.2 Samples are introduced into the gas chromatograph using a purge-and-trap concentrator.
- 7.5.3 Water samples are directly injected into the purging chamber using a 5 mL syringe. Soil samples are extracted in methanol, the methanol extract is mixed with laboratory reagent grade water, and the methanol/water mixture is injected into the purging chamber using a 5 mL syringe.
- 7.5.4 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint \pm three times the standard deviation determined in Section 7.3.
 - 7.5.4.1 Tentative identification of an analyte occurs when a peak from a sample falls within the daily retention time window. Confirmation on a second GC column or by GC/MS analysis may be necessary.
 - 7.5.4.2 Coelution of the m- and p- Xylene isomers may occur.

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7.5.4.3 Validation of GC system qualitative performance must be accomplished by the analysis of mid-level standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

7.5.5 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peak elutions between specified range "marker" compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual check be initially performed, to document proper integration functions.

7.5.6 **When quantifying on a peak area basis by external calibration, collective peak area integration for the fractional ranges must be from baseline (i.e. must include the unresolved complex mixture "hump" areas).** For the integration of individual Target Analytes, surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.

7.5.7 Baseline correction using a system solvent blank is permissible, if conducted in accordance with the procedures and requirements specified in Section 7.7.4.

7.5.8 If the VPH Target Analytes are to be quantitated using this method, and the response for an individual analyte exceeds the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale.

7.5.9 For non-target analytes and target analytes eluting in the aliphatic or aromatic fractions, the upper linear range of the system is based on the highest calibration standard. Refer to Table 3 for concentrations.

7.6 Calculations (external standard)

The concentration of targeted analytes and hydrocarbon ranges in a sample may be determined by calculating the amount of analyte or hydrocarbon range purged, from the peak response using the calibration curve.

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

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The concentrations from the reports are then incorporated with the purge and/or extraction data to arrive at a final concentration.

Water: Concentration ($\mu\text{g/L}$) = $(C)(0.005\text{L})/(V_s)$

Soil: Concentration (mg/Kgdrywt) =
 $(C) (((V_t + ((M/100)(V_o))) (0.005\text{L}) / (V_o)(V_p)(100/100-M)(1/1000))$

Where: C = Concentration calculated by Target in $\mu\text{g/L}$.
V_s = Volume of sample purged in L.
V_o = Weight of sample extracted in Kg.
V_t = Volume of methanol in L.
V_p = Total volume of extract purged in L.
M = Moisture in %.

7.6.2 Required Adjustment of Range Concentration Data: In order to minimize the "double counting" of the same hydrocarbon compounds on both the FID and PID chromatograms, the collective concentrations of MTBE, benzene, toluene, ethylbenzene, and m-, p- and o-Xylene identified on the PID chromatogram must be subtracted from the collective C₅ through C₈ and C₉ through C₁₂ Aliphatic Hydrocarbon concentration value determined using the FID chromatogram.

7.7 Sample Analysis

7.7.1 PID Chromatogram

7.7.1.1 If desired, determine the peak area count for the Target VPH Analytes.

7.7.1.2 Determine the peak area count for the surrogate 2,5-dibromotoluene.

7.7.1.3 Determine the total area count for all peaks eluting 0.1 minutes after the retention time (Rt) for o-Xylene and 0.1 minutes before the retention time for naphthalene.

7.7.1.4 Using the equations contained in Section 7.6.1, calculate the concentrations of the surrogate standard 2,5-dibromotoluene, and C₉ through C₁₀ Aromatic Hydrocarbons. Optionally, calculate the individual concentrations of the Target VPH Analytes.

7.7.2 FID Chromatogram

7.7.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-pentane and 0.1 minutes before

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the Rt for n-nonane. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.7.2.2 Determine the total area count for all peaks eluting 0.1 minutes before the Rt for n-nonane and 0.1 before the Rt for naphthalene. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.7.2.3 Determine the peak area count for the surrogate standard 2,5-dibromotoluene.

7.7.2.4 Using the equations contained in Section 7.6.1, calculate the concentrations of C₅ through C₈ Aliphatic Hydrocarbons, C₉ through C₁₂ Aliphatic Hydrocarbons, and the surrogate standard 2,5-dibromotoluene.

7.7.3 Data Manipulations

7.7.3.1 By definition, the collective concentrations of aliphatic and aromatic fractions of interest **exclude** the individual concentrations of VPH Target Analytes. Accordingly, a series of data manipulation steps are necessary to adjust the collective range concentrations calculated in 7.7.1.4 and 7.7.2.4, to eliminate "double counting" of analytes.

7.7.3.2 Subtract the collective concentration of C₉-C₁₀ Aromatic Hydrocarbons from the collective concentration of C₉-C₁₂ Aliphatic Hydrocarbons.

7.7.3.3 Subtract the individual concentrations of the VPH Target Analytes from the appropriate aliphatic range (i.e., C₅-C₈ or C₉-C₁₂ Aliphatic Hydrocarbons) in which they elute. If the individual concentrations of Target Analytes have not been quantitated, report the values as Unadjusted C₅-C₈ Aliphatic Hydrocarbons and Unadjusted C₉-C₁₂ Aliphatic Hydrocarbons, and indicate "Not Determined" for C₅-C₈ Aliphatic Hydrocarbons and C₉-C₁₂ Aliphatic Hydrocarbons.

7.7.4 Baseline Correction for Instrument Noise Level

7.7.4.1 Range integration areas may be corrected by the automatic subtraction of the baseline established by the injection of a system solvent blank.

7.7.4.2 Instrument baseline shall be established by the purging of a system solvent blank, an air blank, or by activation of a programmed run

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without the injection of any material. All system and run elements and parameters must be identical to those of the sample run.

7.7.4.3 If baseline correction is used, instrument baseline must be established for every batch of samples, and after the analysis of samples that are suspected to be highly contaminated.

7.8 Data Review

7.8.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS/LCSD, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery.
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.9.

7.8.2 Surrogate recovery

All recoveries must meet the method acceptance limits of 70-130%.

The sample is evaluated for recoveries of the surrogate. If the surrogate recovery is outside of the method acceptance limits on one channel but acceptable on the other channel, narrate. If the recovery is high and the sample results are less than the PQL, narrate. If the recovery is low and may be attributable to matrix interference, reanalyze to confirm a matrix effect and narrate. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the soil % moisture is >25% and the surrogate recovery is >10%, narrate. If the soil reanalysis is still low, re-extract.

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7.8.3 Chromatography

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, if the sample contains a concentration of the aliphatic range C₅ through C₈ which was integrated "valley to valley" instead of a "baseline to baseline"), it will be done in Target Review. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration," current revision.

7.8.4 Target Compound Detection

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be crossed out and a GC lab code applied adjacent to its concentration. The possible scenarios are: If an analyte is present on one channel but its concentration is below the PQL, then the analyte and its concentration are crossed out and the GC lab code BDL is written next to the concentration. If an analyte is present but its retention time is ± 0.04 minutes or more than the retention time of the analyte in the preceding CV, then the analyte and its concentration are crossed out and the GC lab code RTW is written next to the concentration.

The GC lab codes are:

BDL - Below Detection Limit

RTW - Retention Time Window

7.9 Reporting

7.9.1 After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, reports, such as chronology or calibration forms, are generated. Reports of Analysis (ROA), LCS/LCSD, MS/MSD and surrogate forms are generated in KIMS. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is

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provided with each package. The final data package from the Organics department is then processed by the Data Management department.

- 7.9.2 The VPH reporting form contains an attestation that indicates whether significant modifications were made to the VPH method, a clear affirmation on whether the QA/QC procedures and standards specified in the method were followed and achieved.
- 7.9.3 Significant modifications may include the use of alternative detectors, the use of other than a purge-and-trap sample preparation, the use of solvents other than those recommended, and the use of a different surrogate than recommended. Any modifications which were made along with any QA/QC deviations are mentioned in the case narrative.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 General Requirements and Recommendations

- 8.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document the quality of data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

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- 8.1.2 A methanol trip blank or acidified laboratory reagent grade water blank should continually accompany each soil sample or water sample batch, respectively, over the course of sampling, storage, and analysis.
- 8.1.3 A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.
- 8.1.4 At a minimum, for each analytical batch (up to 20 samples), an Initial Calibration or opening and closing Calibration Check Standard, Laboratory Method Blank, Laboratory Control Sample (LCS) and LCS duplicate (LCSD) must be run. A duplicate sample (if client requested) and Matrix Spike (MS) and/or MS duplicate (MSD) should be analyzed, at the discretion of the analyst, based upon the nature of the sample. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered. The blank and spiked samples should be carried through all stages of the sample preparation and measurement process.
- 8.1.5 The recommended sequence of analysis is as follows:
- (1) Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration) **[REQUIRED]**
 - (2) Laboratory Method Blank **[REQUIRED]**
 - (3) Laboratory Control Sample and Duplicate **[REQUIRED]**
 - (4) Samples
 - (5) Duplicate sample [if client requested]
 - (6) Matrix Spike/Duplicate [As appropriate]
 - (7) Mid-range Calibration Check Standard [consider after 10 samples, as appropriate]
- 8.1.6 At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, reanalyze the sample.
- 8.2 Minimum Instrument QC
- 8.2.1 The n-pentane (C₅) and MTBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively. This is achievable using the recommended trap and purge-and-trap procedures. Coelution of the m- and p- xylene isomers is permissible. Any surrogates used must be adequately resolved from individual compounds in the VPH Component Standard.

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8.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Section 7.3)

8.2.3 Calibration factors must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of the calibration factors may be assumed if the %RSD over the working range of the curve is less than or equal to 25%. (See Section 7.4)

8.3 Initial and Periodic Method QC Demonstrations

The procedures specified in Section 8.3.1.1 through 8.3.1.3 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

8.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate laboratory reagent grade water and/or clean sand blanks spiked with each analyte of interest at approximately half of the highest calibration standard (100 µg/L water and 16.7-50 mg/Kg soil).

8.3.1.1 Add an appropriate aliquot of the stock or primary dilution standard solution(s) to each of the four replicate laboratory reagent grade water or clean sand blanks. Purge and analyze each replicate according to the procedures described in Section 7.1.

8.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

8.3.1.3 For each analyte, the mean accuracy, expressed as a percentage of the true value, must be between 70% and 130%, and the %RSD must be less than or equal to 25%. Higher recoveries are permissible for n-nonane.

8.4 Ongoing Method QC Demonstrations

8.4.1 Each sample, blank, and Laboratory Control Sample must be spiked with the surrogate spiking solution. Required surrogate recovery is 70% to 130%.

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Recoveries outside of this range must be noted and discussed in the data report form.

8.4.2 At a minimum, with every batch of 20 samples or less the laboratory must analyze the following:

8.4.2.1 **Calibration Check Standard** - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended after the analysis of the tenth sample. If the percent difference (%D) of any analyte within the calibration check standard varies from the predicted response by more than 25%, a new calibration curve must be prepared for that analyte.

8.4.2.2 **Laboratory Method Blank** - A water or soil Laboratory Method Blank is prepared by fortifying a 5 mL laboratory reagent grade water blank with 5 μ L of the surrogate spiking solution, or by fortifying a 15 g sample of clean sand with 1 mL of the surrogate spiking solution. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest.

8.4.2.3 **Laboratory Control Sample and Duplicate**- The Laboratory Control Sample and Duplicate are prepared by fortifying a 5 mL laboratory reagent grade water blank with 10 μ L of the matrix spiking solution, or by fortifying a 15 g sample of clean sand with 500 μ L of the matrix spiking solution. The spike recovery must be between 70% and 130%. RPD is <25%.

8.4.2.4 **Sample duplicate** - Sample duplicates may be laboratory duplicates or field duplicates per the client's request. The %RPD of duplicate samples must not exceed 50%.

8.4.2.5 **System Solvent Blank** - If baseline correction will be employed, as specified in Section 7.7.4, a system solvent blank, air blank, and/or system run must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated.

8.4.3 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) is performed per the client's request.

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8.4.3.1 **Matrix Spike (MS)/MS Duplicate (MSD)** –The water or soil MS/MSD spike is prepared by fortifying an actual 5 mL water sample with 10 μ L of the matrix spiking solution, or by fortifying an actual 15g soil/sediment sample with 500 μ L of the matrix spiking solution.

The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. Lower recoveries of naphthalene are permissible, but must be noted. The %RPD of the MS/MSD should be less than or equal to 25%.

8.4.4 If any of the performance standards specified in Section 8.4.1 and 8.4.2 are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are fallen out must be rerun. If this is not possible, that data must be reported as suspect.

8.5 NCR: Whenever data is not acceptable because of a failing LCS or surrogate recovery or other QC failure, a nonconformance report must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," for procedures on determining the MDL.

Refer to the current revision Method MADEP VPH-04-1.1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", USEPA SW846, third Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Methods 8000B, 8015C, 5030B and the current edition of 8020.

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Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), MADEP, May 2004, Revision 1.1.

Katahdin SOP CA-101, "Equipment Maintenance and Troubleshooting," current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," current revision.

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TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP-VPH-04-1.1

QC check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point external calibration of 15 VPH component standards, and a surrogate. Also, collective calibrations of C ₉ through C ₁₀ aromatic hydrocarbons, C ₅ through C ₈ aliphatic hydrocarbons and C ₉ through C ₁₂ aliphatic hydrocarbons.	Initial calibration prior to sample analysis	The %RSD must be ≤ 25%	Investigate and repeat initial calibration
Initial Calibration Verification (ICV)	Once after each calibration	All analytes ≤ 25 %D of the expected value.	Reanalyze sample Reprepare standard Reprepare standard from fresh stock.
CV	If initial calibration analyzed, daily and after 20 samples, and at end of sequence.	%D for all analytes within ±25%	Evaluate the samples: If the %D >±25% and sample results are < PQL, narrate. If %D >±25% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report samples results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One LCS/LCSD per prep batch	Spike recovery must be between 70% and 130%	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1 (cont.)

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP-VPH-04-1.1

QC check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate	Every sample, blank, and QC sample	Recovery must be between 70% and 130%	Refer to section 7.8.2
Matrix Spike/Matrix Spike Duplicate	Per client request	Recovery must be between 70% and 130%.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample duplicate if requested by the client	One per batch of 20 samples	%RPD of duplicate must be less than 50%.	(1)check calculations for errors (2) Evaluate QC
Demonstration of ability to generate acceptance accuracy and precision using four replicate analyses of a QC check sample	Once per analyst and yearly thereafter	For each analyte, the mean accuracy must be between 70% and 130% and the %RSD must be \leq 25%	Investigate; reprep
MDL and/or LOD/LOQ Verification	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-312-07	METHOD MADEP-VPH-04-1.1
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures		
Procedures	7.5.9 For non-target analytes and target analytes eluting in the aliphatic or aromatic fractions, the upper linear range of the system is based on the highest calibration standard. Refer to Table 3 for concentrations.	See Section 9.5.8
Procedures	7.5.4 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day if after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint \pm three times the standard deviation determined in Section 7.3.	Section 9.5.4 Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint \pm three times the standard deviation determined in Section 9.3.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

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TABLE 3

VPH CALIBRATION CONCENTRATIONS

Component	Level 1 µg/L	Level 2 µg/L	Level 3 µg/L	Level 4 µg/L	Level 5 µg/L	Level 6 µg/L
n-Pentane	5	10	50	100	200	300
2-Methylpentane	5	10	50	100	200	300
MTBE	5	10	50	100	200	300
2,2,4-Trimethylpentane	5	10	50	100	200	300
Benzene	5	10	50	100	200	300
Toluene	5	10	50	100	200	300
n-Nonane	5	10	50	100	200	300
n-Decane	5	10	50	100	200	300
n-Butylcyclohexane	5	10	50	100	200	300
Ethylbenzene	5	10	50	100	200	300
m,p-Xylene	5	10	50	100	200	300
o-Xylene	5	10	50	100	200	300
1,2,4-Trimethylbenzene	5	10	50	100	200	300
Naphthalene	5	10	50	100	200	300
2,5-Dibromotoluene	5	10	50	100	200	300
C ₅ -C ₈	15	30	150	300	600	900
C ₉ -C ₁₂	15	30	150	300	600	900
C ₉ -C ₁₀	5	10	50	100	200	300

Amount of standard added to 5 mL volume:

- Level 1 5 µL of 5 µg/mL standard
- Level 2 10 µL of 5 µg/mL standard
- Level 3 5 µL of 50 µg/mL standard
- Level 4 10 µL of 50 µg/mL standard
- Level 5 4 µL of 250 µg/mL standard
- Level 6 6 µL of 250 µg/mL standard

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 4

HOLDING TIMES AND PRESERVATIVES FOR VPH SAMPLES

Matrix	Container	Preservation	Holding time
Aqueous Samples	40 mL VOC vials w/ Teflon-lined septa screw caps.	Add 3 to 4 drops of 1:1 HCl; cool to 4 (± 2) °C.	14 days
Soil/Sediment Samples	40 mL VOC vials w/ Teflon-lined septa screw caps: add 15 g soil	1 mL methanol for every g soil; add before or at time of sampling; cool to 4 (± 2) °C	28 days

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

TABLE 5

PQLS FOR METHOD MADEP VPH

Parameter/ Method	Analytes	Practical Quantitation Level		
		PQL	PQL	
		(µg/L)	(mg/Kg)	
VPH/MADEP- VPH-04-1.1	Methyl-tert-butylether	5	1.25	
	Benzene	5	1.25	
	Toluene	5	1.25	
	Ethylbenzene	5	1.25	
	m- & p-Xylene	10	2.5	
	o-Xylene	5	1.25	
	Naphthalene	5	1.25	
	C ₅ - C ₈ Aliphatics(FID)	100	25	
	C ₉ - C ₁₂ Aliphatics(FID)	100	25	
C ₉ - C ₁₀ Aromatics(PID)	100	25		

TITLE: METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDRO-CARBONS (MADEP - VPH)

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ (Verbals turned in). DueDate _____

Client:	Primary	Secondary
Method:	Date:	Date:
SDG No: Level:	Initials:	Initials:
KAS No:	Approved : <input type="checkbox"/> Yes	

DODQSM 3.0 DODQSM 4.1 WITH LAB. LIMITS
 QUAPP LAB (REPORT ND's to MDL)

List all curves that are scanned. _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights, TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration (Narrate as needed). _____

Sample I.D's Truncated (NARRATED). YES Please list KAS # below :

**TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR
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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-511-07**, titled **EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP-EPH METHODS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-511-07**, titled **EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP-EPH METHODS**.

Recipient: _____ Date: _____

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

1.0 SCOPE AND APPLICATION

This document describes the protocols of the Massachusetts Department of Environmental Protection Method used for the Determination of Extractable Petroleum Hydrocarbons (EPH), including sample collection, sample extraction and cleanup. The MADEP EPH method is designed to measure the collective concentrations of extractable aliphatic hydrocarbons within the ranges C₉ through C₁₈ and C₁₉ through C₃₆. This method is also designed to measure the collective aromatic hydrocarbons within the C₁₁ through C₂₂ range and to measure the individual concentrations of targeted polynuclear aromatic hydrocarbons (PAHs) in water and soil. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150°C and 265°C. Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, and other petroleum products which contain a significant percentage of hydrocarbons lighter than C₉ or for petroleum products which contain a significant percentage of hydrocarbons heavier than C₃₆.

1.1 Definitions

Extractable Petroleum Hydrocarbons (EPH) - all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Targeted PAH Analytes. EPH is comprised of C₉ through C₁₈ Aliphatic Hydrocarbons, C₁₉ through C₃₆ Aliphatic Hydrocarbons, and C₁₁ through C₂₂ Aromatic Hydrocarbons. EPH concentration data are reported as a toxicologically-weighted summation of the aliphatic and aromatic hydrocarbon fractions.

C₁₁ through C₂₂ Aromatic Hydrocarbons - all aromatic hydrocarbon compounds eluting from naphthalene through benzo(g,h,i)perylene, excluding Targeted PAH Analytes.

C₉ through C₁₈ Aliphatic Hydrocarbons - all aliphatic hydrocarbon compounds eluting from n-nonane to just before n-nonadecane (n-C₁₉).

C₁₉ through C₃₆ Aliphatic Hydrocarbons - all aliphatic hydrocarbon compounds eluting from n-nonadecane through n-hexatriacontane (n-C₃₆).

Targeted PAH Analytes - the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 3.

1.2 Responsibilities

Implementation of this SOP requires sufficiently trained analysts and properly functioning instrumentation. Samples must be properly extracted following Katahdin Analytical Quality Assurance/Quality Control requirements. Only analysts/technicians

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qualified and experienced with this method may perform these procedures. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in the determination of Extractable Petroleum Hydrocarbons to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for EPH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

All standards should be prepared in a hood.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

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Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, as well as cartridges used for the fractionation of EPH samples, should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

A soil or water sample is extracted with methylene chloride, dried with sodium sulfate, solvent exchanged into hexane, and then concentrated using a Kuderna-Danish apparatus. Sample fractionation into aliphatic and aromatic fractions is conducted using a preparative HPLC column and Foxy fraction collector. The two extracts produced are then re-concentrated to final volumes of 1 mL each. The resulting extracts (an aliphatic extract and an aromatic extract) are analyzed separately for EPH using a GC equipped with a flame ionization detector (FID).

3.0 INTERFERENCES

Method interferences are minimized by using high purity reagents and by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone and methylene chloride.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interference will vary from one source to another. A preparative HPLC column cleanup and fractionation procedure is used to overcome many of these interferences, but some samples may require additional cleanup approaches that are beyond the scope of this method.

Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable Petroleum Hydrocarbons. If necessary or desirable, additional sample cleanup or analytical procedures may be employed to minimize or document the presence of such compounds.

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The leaching of plasticizers and other compounds has been observed from commercially available silica gel cartridges used to fractionate EPH sample extracts. Concerns of this nature must be continuously monitored and documented by the analysis of laboratory method blanks.

4.0 APPARATUS AND MATERIALS

- 4.1 1-L amber glass bottles
- 4.2 4 oz. (120 mL) glass wide-mouth jars
- 4.3 Vials: 4 mL glass vials with silicone/PTFE septa in open top caps
- 4.4 Vials: 1.8 mL with silicone/PTFE septa in open-top caps
- 4.5 Glass funnels
- 4.6 2-L Separatory teflon FEP funnels with screw closures
- 4.7 Kuderna-Danish apparatus including 10-mL concentrator tube, 500-mL Evaporative flask and 3-ball Snyder column
- 4.8 250 mL amber bottles with Teflon-lined screw covers
- 4.9 Disposable pipettes: Pasteur, 5 ¾"
- 4.10 25-mL graduated cylinder
- 4.11 1-L graduated cylinder
- 4.12 400-mL beakers
- 4.13 A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples
- 4.14 Nitrogen blowdown apparatus, Organomation N-EVAP
- 4.15 Water bath: heated with a concentric ring cover, capable of temperature control ($\pm 2^\circ$ C). The bath should be used in a hood
- 4.16 Syringes, gas tight, 1.0mL, Hamilton or equivalent, and 10mL Luer-Lok syringe (Popper or equivalent)
- 4.17 Boiling Chips, silicon carbide (carborundum), 12 mesh

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- 4.18 Soxhlet extraction apparatus, or apparatus for equivalent technique such as ASE (accelerated solvent extractor)
 - 4.19 Voa vials, 40 mL
 - 4.20 Filter paper, 18.5 cm diameter
 - 4.21 Wide range pH test strips, CF-type, pH0-14
 - 4.22 Solid Phase Extraction (SPE) cartridges with silica gel (5g/20mL)
 - 4.23 Waters HPLC system, or equivalent, consisting of an autosampler and gradient pump.
 - 4.24 ISCO foxy 200 fraction collector, or equivalent.
 - 4.25 Restek HPLC column, or equivalent, 150mm X 10mm.
-

5.0 REAGENTS

- 5.1 Laboratory reagent grade water: organic free water (Culligan reagent grade water).
 - 5.2 Solvents: hexane, methylene chloride, and acetone; pesticide grade or better.
 - 5.3 Sodium sulfate: (ACS) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray (sodium sulfate may be purchased pre-purified by the manufacturer).
 - 5.4 Sand: free of extractable petroleum hydrocarbons, purified by heating at 400 °C for four hours
 - 5.5 Matrix Spike/Lab Control Sample Spiking Solution: Refer to Table 3
 - 5.6 Surrogate Spiking Solution: 5-alpha androstane and ortho-terphenyl (OTP) at concentrations of 90 ug/mL in acetone
 - 5.7 Fractionation check standard: See Table 3.
 - 5.8 Fractionation surrogate standards: 2-Fluorobiphenyl and 2-Bromonaphthalene at 90 ug/mL in hexane.
 - 5.9 Hydrochloric acid solution (1:1 HCl: H₂O) - slowly add 500 mL of HCl to 500 mL reagent water.
-

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 6.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps.
 - 6.2 Soil and sediment samples are collected in 4 oz. (120 mL) wide-mouth glass jars with Teflon-lined screw caps.
 - 6.3 Sample container, preservation and holding times are summarized in Table 4.
 - 6.4 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the date and time of sampling and any preservatives added.
-

7.0 PROCEDURES

Samples are extracted using methylene chloride, and solvent-exchanged into hexane. The recommended extraction procedure for water samples is a separatory funnel liquid-liquid extraction technique based upon SW-846 Method 3510A. For soil or sediment samples, use of a Soxhlet extraction technique based on SW846 Method 3540C is employed. Alternative extraction procedures are acceptable such as 3541 automated Soxhlet extraction procedure, provided that the laboratory can document acceptable performance. Sonication (3550) may only be used for the extraction of highly contaminated (free product) non-soil/sediments.

WATER EXTRACTION

- 7.1 Mark the meniscus on the 1-liter sample bottle (for later volume determination) and transfer it to a 2-liter separatory funnel.
- 7.2 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, pour 1 liter of laboratory reagent grade water into a 2-liter separatory funnel.
- 7.3 A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS/LCSD, pour 1-liter of laboratory reagent grade water into each of two 2-liter separatory funnels.
- 7.4 A matrix spike/matrix spike duplicate (MS/MSD) is prepared per client request. To prepare MS/MSD, mark the meniscus on each 1-liter sample bottle designated for MS/MSD (for later volume determination) and transfer each to a 2-liter separatory funnel.

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- 7.5 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of EPH surrogate spiking solution using a pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.
- 7.6 To LCS/LCSD and MS/MSD add 1.0 mL of the EPH matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.
- 7.7 Check the pH of the sample with wide-range pH paper. Note the pH in the laboratory notebook. The pH of the sample must be adjusted to pH<2. Adjust all samples and quality control samples as needed with 1:1 HCl.
- 7.8 Add 60 mL methylene chloride to the sample bottle to rinse the inner walls of the container, then add this solvent to the separatory funnel. Add 60 mL methylene chloride directly to separatory funnel for blank, LCS/LCSD.
- 7.9 Seal and shake the separatory funnel vigorously for 3 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, venting should be done immediately after the separatory funnel has been sealed and shaken once.

- 7.10 Allow the organic layer to separate from the water phase for a minimum of 5 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in a 250 mL amber bottle with a Teflon-lined screw-cap.
- 7.11 Repeat the extraction two more times using additional 60 mL portions of solvent. Combine the three solvent extracts in the amber bottle. (Steps 7.5 to 7.10)

CONCENTRATION OF EXTRACTS

- 7.12 For sample volume determination add water to the sample bottle to the level of the meniscus previously marked then transfer this water to a graduated cylinder. Record sample volumes in extraction logbooks to the nearest 10 mLs.
- 7.13 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

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- 7.14 Add approximately 50 mL Hexane directly to the KD body to allow for a solvent exchange.
- 7.15 Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the amber bottle, which contained the solvent extract, with 20 to 30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.
- 7.16 Add one or two clean boiling chips to the K-D flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C). Gently swirl K-D in water until boiling begins. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. . The K-D should be kept in a vertical orientation while on the bath. When the apparent volume of liquid reaches 4 - 6 mLs, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Rinse the Snyder column lower joint with ~ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ~1 mL methylene chloride.
- 7.17 Reduce the hexane extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45°C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipette must be rinsed down at least once or twice with ~1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N₂ sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.18 Concentrate the extract as described in Step 7.17, using hexane to rinse the internal wall of the concentrator tube. Be sure to lower the bath temperature back to 39°C when the solvent exchange is complete. Reduce the extract to 1 mL and place in a 4.0 mL vial. Record the "pre-fractionation" final volume as 1 mL in the extractions logbook.
- 7.19 Record the sample preparation information for the extraction and concentration steps. As a minimum, record the date, sample laboratory number, sample volume, volume and concentration of added surrogates, fractionation surrogates, and matrix spike solutions, and any deviations for problems associated with the extraction of the samples.

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- 7.20 The 1 mL extract is now ready to be cleaned and fractionated using HPLC or SPE fractionation. Proceed to section 7.35.

SOIL EXTRACTION

- 7.21 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

- 7.22 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

- 7.23 Samples should be prepped as follows:

7.23.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.23.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.23.3 Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", for more detailed guidance on subsampling to ensure reproducibility.

- 7.24 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a 10.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 10 g and 20 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

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- 7.25 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 10.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 20 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 20 g sodium sulfate, the method blank must contain 20g in order to evaluate the sodium sulfate as a potential source of contamination.
- 7.26 A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS/LCSD, weigh out two 10.0 ± 0.05 g portions of purified sand in labeled 400 mL beakers. Add 10 g sodium sulfate to each and mix well.
- 7.27 To prepare MS/MSD, weigh out 10 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add between 10 g and 20 g sodium sulfate to each to produce a free-flowing mixture, and mix well.
- 7.28 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.
- 7.29 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of EPH surrogate spiking solution using a pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.
- 7.30 To LCS/LCSD and MS/MSD add 1.0 mL of the EPH matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.
- 7.31 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the flat-bottom flasks when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 45-50% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18 ± 2 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

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- 7.32 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.
- 7.33 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.
- 7.34 See sections 7.13 through 7.20 for the concentration of the extracts.

Fractionation

NOTE: If using the SPE cartridges: The Fractionation step is a critical yet highly sensitive procedure. Small changes in the volumes of eluting solvents, fractionation equipment, and/or fractionation techniques can significantly impact the proportion of hydrocarbons segregated in either the aliphatic or aromatic fractions. Considerable care and attention is required to ensure satisfactory results.

- 7.35 Fractionation checks are analyzed by HPLC on a daily basis before any samples are fractionated. For SPE cartridges: fractionation checks are run with each new lot of SPE cartridges or every three months. A fractionation check is made by adding 1mL fractionation check solution to the previously concentrated 1 mL extract. The fractionation checks are labeled with the lot number and date of fractionation. These checks are to be performed before sample fractionation to confirm the amount of hexane necessary to prevent aromatic breakthrough into the aliphatic fraction.
- 7.36 Add 1.0 mL of the fractionation surrogate spiking solution with a gas tight syringe to the 1 mL hexane extract.

HPLC FRACTIONATION

- 7.37 Make sure the methylene chloride and hexane bottles are full. All solvents need to be filtered and sonicated for at least 15 minutes prior to putting on the HPLC.
- 7.38 Turn pump on at 1.00 mL/min by pressing the home button and then typing 1.00 and enter. Make sure the solvent flowing is hexane. The %A under the flow rate should

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be at 100%. If it is not hit enter until the %A line is highlighted and type in 100 then enter until sparge is highlighted.

- 7.39 Next prime the pumps.
- 7.40 Take a 5 mL disposable syringe and connect to the prime valve on the front bottom of the pump. Turn knob until it reads draw and then draw the syringe fairly slowly. You should see bubbles coming through the clear tubing into the syringe. Repeat at least three more times or until there is no more air coming through the lines.
- 7.41 Repeat 7.40 for the methylene chloride . You must put solvent B at 100% before priming.
- 7.42 When finished priming change pump back to 100% hexane following 7.38.
- 7.43 Let equilibrate for about 5 minutes then purge the autosampler.
- 7.44 At the autosampler, on the front screen, hit the purge page button and then start purge. The settings are already there for an 8 minute purge.
- 7.45 When the purge is finished go to the pump hit the home button and change the flow rate to 4.00 mL/min. Let equilibrate for at least 5 minutes.
- 7.46 At the pump push the program table button and select table 1. Parameters are shown in table 5.
- 7.47 Now push the program method button and scroll over to enter the starting and ending vial numbers that correspond to the samples that are in the autosampler.
- 7.48 Push the operate methods button twice and wait for the start run prompt at the bottom of the screen.
- 7.49 Turn on the Isco Foxy 200 fraction collector using the power button.
- 7.50 Load the Foxy tray with 40 mL VOA vials. You will need two vials per sample, the first being for the aliphatic and the second for the aromatic. Start with position one and put a waste VOA vial after every aromatic fraction due to the fact the collector skips a space after every injection.
- 7.51 After the vials are on the tray push the run A button on the front of the Foxy. The sampling arm should move to the ready position waiting for the injection to occur.
- 7.52 Push the start run button on the pump which will prompt the autosampler to start the injection. Most injection are 1.0 mL, however you can inject a smaller volume as long as you make sure there is enough sample in the vial to inject. When the injection occurs the sampling arm of the Foxy will position itself over the aliphatic

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

vial and start collecting. When the aliphatic portion finishes the arm automatically moves to the second vial and immediately starts collecting the aromatic fraction.

- 7.53 The total run time for one sample is 16.50 minutes followed by a 6 minute equilibration time in hexane.
- 7.54 Place all fractions on the N-evap once the fractionation is complete. The aliphatic fraction should be transferred into a 12 mL conical vial. The aromatic fraction should be left in the VOA vial until it reaches a suitable volume to transfer into a 12 mL conical vial.
- 7.55 Blow all fractions down to the volume at which they were injected (if 1.0 mL was injected, the final volume will be 1 mL, if 0.5 mL was injected the final volume will be 0.5 mL). All aliphatic fractions are in Hexane, while all aromatic fractions are in methylene chloride.
- 7.56 Transfer all samples to a 2.0 mL screw top vial.
- 7.57 Samples are now ready for analysis.
- 7.58 All samples fractionated on the HPLC should be written in the HPLC03 logbook (figure 2).

SPE CARTRIDGE FRACTIONATION

- 7.59 Place SPE cartridges on the sample collection apparatus. With the valve under the sample in the open position, rinse each SPE cartridge with 30mL of hexane, using a 10mL syringe. Close the valve when approximately 0.5cm of solvent remains above the cartridge frit. Do not allow the silica gel cartridges to dry out after the solvent has passed through. Empty the collection apparatus of eluted solvent.
- 7.60 Place a 40mL VOA vial, labeled as the aliphatic fraction, under each SPE cartridge in the sample collection apparatus. Add 1mL of the fractionation check or sample to the top of the SPE cartridge. Open the valve. Just as the sample/solvent layer reaches the frit, add ~17mL of hexane by syringe. (The amount of hexane is determined by the fractionation check done on each lot or every three months.) The solvent should be added slowly, however, the cartridge must not be allowed to go dry. Rinsing the sides of the cartridge when adding the hexane may help improve recoveries. Close the valve when approximately 0.5cm of solvent remains. Remove the test tubes.
- 7.61 Place a new 40mL VOA vial, labeled as the aromatic fraction, under the sample collection apparatus. Open the valve. Just as the hexane layer reaches the frit, add 20mL of methylene chloride, by syringe, to collect the aromatic fraction. Rinsing the

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sides of the cartridge when adding the methylene chloride may help improve recoveries. Remove the test tubes.

- 7.62 All fractions should be placed on the N-EVAP bath and blown down to the volume at which they were fractionated (if 1.0mL was fractionated, the final volume will be 1mL). The final volume recorded in the logbook and computer, however, should be 2.0 mL since this is what the actual final volume is. All aliphatic fractions are in hexane, while all aromatic fractions are in MeCl₂.
- 7.63 Record the sample preparation information for the fractionation and concentration steps. At a minimum, record the SPE cartridges lot#, fractionation date, fractionation initials, volumes of hexane and methylene chloride added for each fraction, concentration volume, concentration initials, and any deviations or problems associated with the fractionation of the samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) is required for each and every item listed below:

- Each sample matrix
- Each extraction method
- Every extraction batch of twenty or fewer samples

Sample specific matrix spikes (MS) and matrix spike duplicates (MSD) are extracted per client request or per project requirements.

Refer to Katahdin SOP CA-322, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH), current revision, for other Quality Control parameters and acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to Katahdin SOP CA-322, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH), current revision.

10.0 APPLICABLE DOCUMENTS/REFERENCES

**TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR
 ANALYSIS BY MADEP - EPH METHODS**

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004, revision 1.1.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Office of Solid Waste and Emergency Response, U.S. EPA

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

Katahdin Analytical Services, Inc. SOP CA-322, current revision, Method for the Analysis of Extractable Petroleum Hydrocarbons (MADEP - EPH)

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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Figure 1 Example of Extraction Logbook Page

Figure 2 Example of HPLC03 Logbook Page

Figure 3 Example of HPLC 03 Maintenance Logbook Page.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Prep for waters and soils for EPH determination	Method blank	One per prep batch or 20 samples whichever is more frequent.	Refer to analytical method.	Refer to analytical method.
	LCS/LCSD	One each per prep batch	Refer to analytical method.	Refer to analytical method.
	Matrix Spike/Matrix Spike Duplicate	Per client request	Refer to analytical method.	Refer to analytical method.
	Sample Duplicate/ Matrix Spike	Per client request	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst performing the method, then yearly	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL and-or LOD/LOQ verification study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-511-07	MADEP METHOD, current revision
Apparatus/Materials	Automated HPLC and Fraction Collector	SPE cartridges
Reagents		
Sample preservation/ handling		
Procedures	<ol style="list-style-type: none"> 1) Initial solvent exchange with addition of 50 mLs hexane to KD body prior to sample addition, and solvent exchange a second time into hexane via the addition of 10 mLs of hexane during the nitrogen blowdown. 2) 7.15 ...with 20 to 30 mL of methylene chloride ...to complete the quantitative transfer. 3) 7.16... Place the K-D apparatus on a hot water bath (80-90°C). Gently swirl K-D in water until boiling begins. 4) Fractionate samples using HPLC. 	<ol style="list-style-type: none"> 1) Addition of 50 mLs of hexane to KD body after concentration of extract in methylene chloride to 1mL to accomplish solvent exchange. 2) 9.1.2.5 ...with 100 to 125 mL of methylene chloride to complete the quantitative transfer. 3) 9.1.10 Place the K-D apparatus on a hot water bath (80-90 °C) so that... 4) Fractionate samples with SPE cartridges.
QC - Spikes	<ol style="list-style-type: none"> 1) Surrogate, spike and fractionation surrogate all prepared at 90 ug/mL for use in a 2 mL or prefractionated volume. 2) Fractionation check solution: aliphatics, aromatics, 5-alpha androstane, and o-terphenyl at 200ug/mL each 	<ol style="list-style-type: none"> 1) Surrogate at 40 ug/mL, matrix spike at 50-150 ug/mL, fractionation surrogate at 40 ug/mL. 2) Fractionation check solution: aliphatics and aromatics at 200ug/mL each
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

TABLE 3

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, AND MATRIX SPIKE CONCENTRATIONS - MADEP - EPH METHOD

ANALYTE	Stock * Standards (ng/uL)	Fractionation** Check Solution (ng/uL)	Matrix Spike Standard *** (ng/uL)
Naphthalene	1,000	200	90
2-Methylnaphthalene	1,000	200	90
Acenaphthylene	1,000	200	90
Acenaphthene	1,000	200	90
Fluorene	1,000	200	90
Phenanthrene	1,000	200	90
Anthracene	1,000	200	90
Fluoranthene	1,000	200	90
Pyrene	1,000	200	90
Benzo(a)Anthracene	1,000	200	90
Chrysene	1,000	200	90
Benzo(b)Fluoranthene	1,000	200	90
Benzo(k)Fluoranthene	1,000	200	90
Benzo(a)Pyrene	1,000	200	90
Indeno(1,2,3-cd)Pyrene	1,000	200	90
Dibenzo(a,h)Anthracene	1,000	200	90
Benzo(g,h,i)Perylene	1,000	200	90
o-Terphenyl (surr)	1,000	200	90
Nonane	1,000	200	90
Decane	1,000	200	90
Dodecane	1,000	200	90
Tetradecane	1,000	200	90
Hexadecane	1,000	200	90
Octadecane	1,000	200	90
Nonadecane	1,000	200	90
Eicosane	1,000	200	90
Docosane	1,000	200	90
Tetracosane	1,000	200	90
Hexacosane	1,000	200	90
Octacosane	1,000	200	90
Triacontane	1,000	200	90
Hexatriacontane	1,000	200	90
5-alpha androstane(surr)	1,000	200	90
Petroleum Reference Std	1,000	-----	-----

* The Aromatic Hydrocarbon Stock Standards (17 PAH compounds and o-terphenyl) should be prepared in methylene chloride. The Aliphatic Hydrocarbon (consisting of 14 normal alkanes and 5-alpha androstane) should be prepared in hexane.

** The Fractionation Check Standard should be prepared in hexane.

**TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR
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*** The Matrix Spike Solution should be prepared in acetone. The o-terphenyl and 5-alpha androstane surrogate solution should also be prepared in acetone.

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TABLE 4

HOLDING TIMES AND PRESERVATIVES FOR EPH SAMPLES

MATRIX	CONTAINER	PRESERVATION	HOLDING TIME
Aqueous Samples	1 Liter amber glass bottle with Teflon-lined screw cap	Add 5 mL of 1:1 HCl; cool to 4 (±2) °C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap	Cool to 4 (±2) °C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap. Jar should be filled to only 2/3 capacity to avoid breakage if expansion occurs during freezing.	Freeze at -10 °C in the field or in the laboratory*	Samples must be extracted within 14 days of the date thawed and extracts analyzed within 40 days of extraction*

*Samples processed in the laboratory must be preserved at 4 (±2) °C and frozen within 48 hours of the time of collection. Frozen samples may be held for up to one year prior to analysis and must be extracted within 24 hours of thawing.

**TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR
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TABLE 5
HPLC PROGRAM 1 RUN PARAMETERS

RUN TIME(Min)	FLOW RATE (mL/min)	%A (Hexane)	%B MeCl ₂	CURVE #
Initial	4.00	100	0	*
3.00	4.00	0	100	11
10.50	4.00	100	0	11
16.50	4.00	100	0	11
22.50	0.00	100	0	11

**TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR
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TABLE 6

ISCO FOXY 200 FRACTION COLLECTOR

- Use the time windows function
- Collect Vial 1 from 0.00 min – 3.00 min
- Collect Vial 2 from 3.00 min – 10.50 min
- Total fraction size is 10.50 min
- There is a 6 minute equilibration time in hexane where it diverts the hexane to waste until the new injection.

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

EXAMPLE OF EXTRACTION LOGBOOK PAGE

EPH Sep

**KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - MADEP EPH**

Extraction Method:	SW846 3520 (CLLE)	SW846 3510 (SEP) ✓	SW846 3540 (SOX)	SW846 3545 (ASE)
Surrogate ID:	G-6733/6664	Spike ID: 44226	Frac. Surrogate ID: G-6734	
MeCL2 Lot #	H49458	Hexane Lot # H43609	NaSO4 Lot # 1744004	HCL Lot #
KD Filter Paper Lot #	R11472346	Nitrogen Water Bath Temp.		

Date Extracted	Ext. Vol.	Sample ID	Wt/Vol (g/ml)	Surr. Vol.	Spike Vol.	Final Vol. pre-Frac.	Date Conc. pre-Frac.	Conc. in Pre-Frac.	Tray Location	Frac. Vol.	Frac. Surr. Vol.	Final Vol. Post-Frac.	Date Conc. post-Frac.	Tray Location	Conc. in Post-Frac.	Comments
4/5/10	100	W435453-1	1000	1mL	NR	1mL	4/5/10	100	EPH101 B6	1mL	2mL	4-9-10	EPH	KT	R123344	
↓	↓	-2	↓	↓	↓	↓	↓	↓	B7	↓	↓	↓	↓	E5	↓	
↓	↓	-3	↓	↓	↓	↓	↓	↓	B8	↓	↓	↓	↓	E6	↓	
4-9-10																

Date Extracted	Ext. Vol.	Sample ID	Wt/Vol (g/ml)	Surr. Vol.	Spike Vol.	Final Vol. pre-Frac.	Date Conc. pre-Frac.	Conc. in Pre-Frac.	Tray Location	Frac. Vol.	Frac. Surr. Vol.	Final Vol. Post-Frac.	Date Conc. post-Frac.	Tray Location	Conc. in Post-Frac.	Comments
4/5/10	100	SD1390-2E	1020	1mL	NR	1mL	4/6/10	105	EPH101 B7	1mL	2mL	4-9-10	EPH	KT		
↓	↓	-4E	1010	↓	↓	↓	↓	↓	B10	↓	↓	↓	↓	E8	↓	
↓	↓	-6B	1010	↓	↓	↓	↓	↓	B11	↓	↓	↓	↓	E9	↓	
↓	↓	-8B	1050	↓	↓	↓	↓	↓	B12	↓	↓	4-10-10	↓	E10	JUP	
↓	↓	-10B	1060	↓	↓	↓	↓	↓	C1	↓	↓	↓	↓	E11	A1	
↓	↓	-12B	1060	↓	↓	↓	↓	↓	C2	↓	↓	↓	↓	E12	A2	
↓	↓	SD1391-6A	1060	↓	↓	↓	↓	↓	C3	↓	↓	↓	↓	A3	↓	
JUP-4-10-10																

Reviewed By: _____ Date: _____

FIGURE 2

TITLE: EXTRACTION OF PETROLEUM HYDROCARBONS FROM SAMPLES FOR ANALYSIS BY MADEP - EPH METHODS

EXAMPLE OF HPLC03 LOGBOOK PAGE

Katahdin Analytical Services
HPLC03 Logbook

Analytical Method:	MA-EPH: <i>X</i>	Other:	Flow: 4.0 ml/min.
MeCl2 Lot #:	<i>H49E58</i>	Hexane Lot #:	<i>H47E07</i>

Tray Pos. No.	Sample Identification	Date	Initials	Inj. Volume	Dilution	Comments
<i>2</i>	<i>SS-13</i>	<i>3-29-10</i>	<i>JUP</i>	<i>1 mL</i>	<i>1</i>	
<i>3</i>	<i>↓ -14</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>NO CAP</i>
<i>3</i>	<i>↓ -15</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>3</i>	<i>↓ -16</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>
<i>1</i>	<i>FC-17</i>	<i>3-31-10</i>	<i>JUP</i>	<i>1 mL</i>	<i>1</i>	
<i>2</i>	<i>W675404-1</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>3</i>	<i>↓ -2</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>4</i>	<i>↓ -3</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>5</i>	<i>W6-75297-4</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>Filtered</i>
<i>6</i>	<i>↓ -5</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>7</i>	<i>SD1390-2</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>8</i>	<i>↓ -4</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>9</i>	<i>↓ -8</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>10</i>	<i>↓ -6</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>11</i>	<i>↓ -10</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>12</i>	<i>↓ -12</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>13</i>	<i>FC-18</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>1</i>	<i>FC-19</i>	<i>4-1-10</i>	<i>JUP</i>	<i>1 mL</i>	<i>1</i>	
<i>2</i>	<i>W675404-4</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>Filtered</i>
<i>3</i>	<i>↓ -5</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>4</i>	<i>SD1391-6</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>5</i>	<i>SD1425-3</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>6</i>	<i>↓ -4</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>7</i>	<i>W675456-1</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	
<i>8</i>	<i>↓ -2</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	<i>↓</i>	

JUP 03/10

Reviewed by:
EX-011 - Revision 1 - 03/16/2010

Review Date:
QAEX188

000006

FIGURE 3

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS (MADEP - EPH)

Prepared By: Peter Lemay Date: 6/98

Approved By: _____

Group Supervisor: Peter Lemay Date: 2/12/01

Operations Manager: John C. Benton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2.12.01

General Manager: Dennis P. Keefe Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, other updates to sections 7, 8 and QA Table.	DN	2.12.01	2/12/01
02	Changes to sections 2.2, 5.2.2, 7.3.1.3, 7.5.1.1, 7.6.3.4, and 8.0. Also changes to tables 2, 3, and 4	DN	5.2.02	5.2.02
03	Added definitions and information for new data processing system. Added or changed wording to clarify section 7 and Table 2. Added wording to sections 8 + 9 per recent NELAP + Navy audit responses. Minor changes throughout. New figures	MRC	11.15.04	11.15.04
04	Changed terminology for LCS:MS's. Corrected Soil H.T. Added required LCSD and breakthrough check. updated reference. Included KIMS forms. minor changes throughout	LAD	05/30/05	05/30/05
05	Changed references from MADEP EPH-98-1 to MADEP EPH-04-1 Fixed typographical and grammatical errors	LAD	04/06	04/06

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
(MADEP - EPH)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Many changes made throughout, including but not limited to, adding linear regression calibration, spike concentrations, % RSD criteria, PQL's, standard solvent, freezing of soil samples. Refer to QAM/SOP change form filed w/ SOP in QA for more details.	LAD	09/07	09/07
07	Sect. 2.1: Changed solvent from hexane to methylene chloride and added solvent exchange step.	LAD	03/08	03/08
08	Changed surrogate from 1-chloro-octadecane to squalene. Added HPLC fractionation references. Updated MDL/LOD/LOQ verification criteria. Updated references. Updated Logbook page and Data Review checklist.	LAD	04/10	04/10
09	Added ICV criteria. Removed RSD. Added % D. Difference. LAD 06/07/10 Added Average Calibration Model to sections 7.3, 7.5 and Table 1.	LAD	06/10	06/10

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-322-09**, titled **METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS MADEP - EPH METHOD**.

Recipient: _____ Date: _____

**KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE**

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**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

1.0 SCOPE AND APPLICATION

This method is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C₉ through C₁₈, and C₁₉ through C₃₆. Extractable aromatic hydrocarbons are collectively quantitated within the C₁₁ through C₂₂ range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 265 °C.

This method is based on a solvent extraction, HPLC or silica gel cartridge fractionation process, and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

This method is also able to measure the individual concentrations of Target Polynuclear Aromatic Hydrocarbons (PAH) Analytes, including Diesel PAH Analytes, in water and soil.

The fractionation step described in this method may be eliminated to allow for the determination of a Total Petroleum Hydrocarbon (TPH) value, and/or obtain qualitative "fingerprinting" information. While TPH provides little information on the chemistry, toxicity, or environmental fate of petroleum mixtures, it may be a cost-effective screening tool in cases where a relatively low concentration of contamination is suspected.

Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, or other petroleum products that contain a significant percentage of hydrocarbons lighter than C₉. This method, in and of itself, is also not suitable for the evaluation of petroleum products that contain a significant percentage of hydrocarbons heavier than C₃₆.

Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target PAH Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target or Diesel PAH Analyte. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analysis by dissimilar columns, gas chromatography/mass spectrometry (GC/MS) analysis, or other suitable technique is recommended in cases where a target PAH analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where coelution of a non-targeted hydrocarbon compound is suspected.

This is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance. MADEP encourages methodological innovations which (a)

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innovations which (a) better achieve method and/or data quality objectives, (b) increase analytical precision and accuracy, (c) reduce analytical uncertainties and expenses, and/or (d) reduce the use of toxic solvents and generation of hazardous wastes. Laboratories that modify this method must achieve all required performance and acceptance standards, and must have on file a Standard Operating Procedure which thoroughly describes the revised or alternative method, and documentation which demonstrates an equivalent or superior level of performance. All significant modifications to the method must be disclosed and described on the data report form.

1.1 Definitions

Aliphatic Hydrocarbon Standard is defined as a 14 component mixture of the normal alkanes listed in Table 3. The compounds comprising the Aliphatic Hydrocarbon Standard are used to (a) define and establish windows for the two aliphatic hydrocarbons ranges, and (b) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.

Analytical Batch is defined as a group of field samples with similar matrices that are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

Aromatic Hydrocarbon Standard is defined as a 17 component mixture of the polynuclear aromatic hydrocarbons (PAHs) listed in Table 3. The compounds comprising the Aromatic Hydrocarbon Standard are used to (a) define the individual retention times and chromatographic response factors for each of the PAH analytes listed in Table 3, (b) define and establish the window for the C₁₁ through C₂₂ Aromatic Hydrocarbon range, and (c) determine an average chromatographic response factor that can in turn be used to calculate the collective concentration

C₉ through C₁₈ Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds eluting from n-nonane (n-C₉) to just before n-nonadecane (n-C₁₉).

C₁₉ through C₃₆ Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds eluting from n-nonadecane (n-C₁₉) through n-hexatriacontane (n-C₃₆).

C₁₁ through C₂₂ Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds eluting from naphthalene through Benzo(g,h,i)Perylene, excluding Target PAH Analytes.

Calibration Check Standard is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock standard solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

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Calibration Standards are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

Diesel PAH Analytes are defined as naphthalene, 2-methylnaphthalene, phenanthrene, and acenaphthene, and are a subset of Target PAH Analytes. For most sites contaminated by a release of (only) diesel or #2 fuel oil, Diesel PAH Analytes will be the only Target PAH Analytes of interest.

Extractable Petroleum Hydrocarbons (EPH) are defined as collective fractions of hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. EPH is comprised of C₉ through C₁₈ Aliphatic Hydrocarbons, C₁₉ through C₃₆ Aliphatic Hydrocarbons, and C₁₁ through C₂₂ Aromatic Hydrocarbons.

Field Duplicates are defined as two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

Fractionation Surrogate Standards are compounds that are spiked into the sample extract immediately prior to fractionation, in order to determine if significant quantities of naphthalene or substituted naphthalenes are being stripped into the aliphatic extract and to evaluate fractionation efficiency.

Instrument Thermal Bleed is defined as a programmed run without the injection of any material. The purpose of the Instrument Blank is to determine the level of noise and baseline rise attributable solely to the GC system, in the absence of any other analytes or system contaminants.

Laboratory Control Sample (LCS) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution. The LCS is prepared and analyzed in the same manner as the samples and its purpose is to determine the bias of the analytical method.

Laboratory Control Sample Duplicate (LCSD) is defined as a laboratory reagent grade water blank or clean sand blank fortified with a matrix spiking solution, processed and analyzed in the same manner as the LCS. The analysis of the LCSD gives a measure of the precision associated with laboratory procedures, but not with sample collection, preservation or storage procedures.

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Matrix Duplicates (DUP) are defined as duplicate samples prepared and analyzed separately with identical procedures. For soils samples DUPs are taken from the same sampling container; for aqueous samples, a second sample container is used. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

Matrix Spike Sample (MS) is defined as an environmental sample that has been spiked with a matrix spiking solution containing known concentrations of method analytes. The MS sample is treated and analyzed exactly as other samples, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of an unspiked sample aliquot. The measured values in the MS sample must be corrected for background concentrations when calculating recoveries of spiked analytes.

Laboratory Method Blank is defined as an aliquot of laboratory reagent grade water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

Matrix Spiking Solution is defined as a solution prepared independently from the calibration standards, containing known concentrations of method analytes.

Surrogate Standards are compounds spiked into all samples, blanks, and matrix spikes to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

Target PAH Analytes are defined as the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 3.

Total Petroleum Hydrocarbons (TPH) are defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. TPH is equivalent to the summation of C₉ through C₁₈ Aliphatic Hydrocarbons, C₁₉ through C₃₆ Aliphatic Hydrocarbons, and C₁₁ through C₂₂ Aromatic Hydrocarbons.

Unadjusted C₁₁ through C₂₂ Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds eluting from naphthalene through benzo(g,h,i)Perylene.

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Unadjusted TPH is defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, including the target PAH analytes.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect, chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of EPH by MADEP EPH-04-1. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of EPH by MADEP EPH-04-1 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

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Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS (material safety data sheet) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. EPH sample vials are considered "P" waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

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- 2.1 A sample submitted for EPH analysis is extracted with hexane, dried over sodium sulfate, and concentrated in a Kuderna-Danish apparatus. Sample separation into aliphatic and aromatic fractions is conducted using HPLC fractionation. The two extracts produced are then re-concentrated to final volumes of 1mL each (i.e., an aliphatic extract and an aromatic extract). The extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively integrated within the C₉ through C₁₈ and C₁₉ through C₃₆ ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C₁₁ through C₂₂ range, and is (optionally) used to identify and quantitate individual concentrations of Target PAH Analytes.
- 2.2 Average calibration factors, determined using an aliphatic hydrocarbon standard mixture, are used to calculate the collective concentrations of C₉ through C₁₈ and C₁₉ through C₃₆ aliphatic hydrocarbons. An average calibration factor determined using a PAH standard mixture is used to calculate a collective C₁₁ through C₂₂ aromatic hydrocarbon concentration. Calibration factors determined for individual components of the PAH standard mixture are also used to calculate individual concentrations of Target PAH Analytes.
- 2.3 This method is suitable for the analysis of waters, soils, and sediments.
- 2.4 This method is based on (1) USEPA Methods 8000, 8100, and 3630, SW-846, "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, III, IIIA, and IIIB 1996, 1998 & 2004.; (2) Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990; (3) "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992; and (4) Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004, revision 1.1.

3.0 INTERFERENCES

- 3.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride.
- 3.2 High purity reagents must be used to minimize interference problems.
- 3.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a system solvent blank to check for cross-contamination.

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- 3.4 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled.
- 3.5 Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable and Total Petroleum Hydrocarbons.
- 3.6 Because of their weakly polar nature, naphthalene and substituted naphthalenes are readily fractionated into the aliphatic extract. By using HPLC fractionation this contamination is eliminated. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the evaluation of the aliphatic fraction for the presence of naphthalene and 2-methylnaphthalene in the LCS/LCSD pair on a batch basis. The fractionation surrogate, 2-Bromonaphthalene, is used to monitor sample-specific fractionation efficiency.

4.0 APPARATUS AND MATERIALS

- 4.1 The following glassware is used for this method:
- 4.1.1 auto sampler: 2mL glass vials with Teflon-lined rubber crimp caps
 - 4.1.2 10mL vials with Teflon-lined caps
 - 4.1.3 Class "A" volumetric flasks: 10, 25, 50 and 100mL
 - 4.1.4 Class "A" volumetric pipettes: 1, 5 or 10mL
- 4.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1g must be used for weighing soil samples.
- 4.3 Gas Chromatograph
- 4.3.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph for use with capillary columns is required. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. The current system is a Hewlett Packard 5890

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GC connected to the Turbochrom data acquisition system, which is connected to the Target data processing system.

4.3.2 Current columns are: Primary aromatic analysis – ZB-5 30M x 0.53mm id x 1.5µm film thickness. Secondary aromatic or aliphatic analysis – ZB-1 30M x 0.53mm id x 1.0µm film thickness

4.3.3 Detector: A Flame Ionization Detector (FID) is required.

4.3.4 Auto sampler: An auto sampler capable of making 1 to 4 µL injections is recommended.

4.4 Microsyringes: 10-µL, 100-µL, 250-µL, 500-µL, 1000-µL

5.0 REAGENTS

5.1 Reagents- Solvents: hexane, methylene chloride, and acetone; pesticide grade or better. Store reagents away from other solvents.

5.2 Stock Standard Solutions

Stock standard solutions at approximately 1000ng/µL are purchased as certified solutions.

5.2.1 Aromatic Hydrocarbon Standard: The Aromatic Hydrocarbon Standard consists of the 17 PAH compounds listed in Table 3, a surrogate compound, and fractionation surrogate compounds in methylene chloride.

5.2.2 Aliphatic Hydrocarbon Standard: The Aliphatic Hydrocarbon Standard consists of the 14 normal alkanes listed in Table 3 and a surrogate compound in methylene chloride.

5.2.3 Petroleum Reference Standard: The Petroleum Reference Standard consists of an API or commercial diesel fuel. Prepare stock standard solutions by accurately weighing approximately 0.0100g of neat product. Dissolve neat product in hexane and dilute to volume in a 10mL volumetric flask.

5.3 Surrogate Standards

5.3.1 Surrogate standards are used to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

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- 5.3.2 The recommended surrogate standards are 5-alpha androstane and ortho-terphenyl (OTP) which are available from Restek or similar supplier.
 - 5.3.3 The surrogate standard 5-alpha androstane is purchased as a certified solution in hexane. This solution is added to the Aliphatic Hydrocarbon standard.
 - 5.3.4 The surrogate standard OTP is purchased as a certified solution in methylene chloride. This solution is added to the Aromatic Hydrocarbon standard.
 - 5.3.5 Surrogate Spiking Solution: The recommended surrogate spiking solution is comprised of a mixture of the COD and 5-alpha androstane surrogate standards. Prepare a surrogate spiking solution that contains the surrogate standards at a concentration of 90ng/μL in acetone. Each sample, blank, and matrix spike is fortified with 1.0mL of the surrogate spiking solution.
- 5.4 Fractionation Surrogate Standards
- 5.4.1 The fractionation surrogate standards are added to the sample (hexane) extract just prior to fractionation. The purpose of the fractionation surrogate standards is to monitor the efficacy of the fractionation process, and ensure that unacceptable quantities of naphthalene and substituted naphthalenes are not being stripped into the aliphatic extract.
 - 5.4.2 The recommended fractionation surrogate standards are 2-Fluorobiphenyl and 2-Bromonaphthalene. Alternative fractionation surrogate compounds are permissible, provided that a demonstration is made that such compounds exhibit polarities/fractionation properties similar to naphthalene.
 - 5.4.3 The fractionation surrogate standards are purchased as certified in Methylene Chloride. This solution is added to the Aromatic Hydrocarbon standard.
 - 5.4.4 Fractionation Surrogate Spiking Solution: The recommended fractionation surrogate spiking solution is comprised of 2-Fluorobiphenyl and 2-Bromonaphthalene prepared in hexane at concentrations of 90ng/μL. An aliquot of 1mL of the fractionation surrogate spiking solution is added to the 1mL EPH sample extract prior to silica gel fractionation.
- 5.5 Matrix Spike Standard

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- 5.5.1 Analytes from each hydrocarbon group (i.e., aromatic and aliphatic hydrocarbons) are used in a matrix spiking solution, which is prepared independently from the calibration standards.
- 5.5.2 The recommended spiking solution, consisting of all 14 normal alkanes and 17 PAHs as listed in Table 3, is prepared in acetone at concentrations of 90ng/ μ L each. Each selected matrix spike as well as laboratory control/laboratory control duplicate sample is fortified with 1.0mL of the matrix spike standard.
- 5.6 Fractionation Check Solution
 - 5.6.1 The Fractionation Check Solution is used to monitor the fractionation efficiency of the silica gel cartridge, and establish the optimum volume of hexane needed to sufficiently elute aliphatic hydrocarbons, but not strip aromatic hydrocarbons.
 - 5.6.2 Prepare a Fractionation Check Solution in hexane containing 200ng/ μ L of the Aliphatic Hydrocarbon standard (C₉-C₃₆ alkanes), Aromatic Hydrocarbon standard (targeted PAH analytes), and extraction surrogates o-terphenyl and 5-alpha androstane. The final solution will contain 14 alkanes, 17 PAHs, and extraction surrogates at concentrations of 200ng/ μ L each. Alternative concentrations are permissible.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 6.1 Aqueous samples are collected in 1 liter amber glass bottles with Teflon-lined screw caps.
- 6.2 Soil and sediment samples are collected in wide-mouth glass jars with Teflon-lined screw caps.
- 6.3 Aqueous samples must be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5mL of 1:1HCl to a 1 liter sample. The use of alternative acids is permissible. Following collection and addition of acid, the sample must be cooled to 4°C (\pm 2°C).
- 6.4 Soil and sediment samples must be cooled to 4°C (\pm 2°C) immediately after collection.
- 6.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

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- 6.6 Aqueous samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.
- 6.7 Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

A summary of sample collection, preservation, and holding times is provided in Table 1.

Refer to Katahdin SOP CA-511, Extraction of Petroleum Hydrocarbons from Samples for Analysis by MADEP - EPH Methods, current revision.

7.0 PROCEDURES

7.1 GC Conditions

Refer to instrument logbook for the current column and conditions.

Typical conditions are:

- Oven Program:
 - Set oven temperature to 70°C
 - then 10°C/min to 190°C
 - then 6°C/min to 238°C
 - then 2.5°C/min to 310°C and hold for 10 minutes.
 - Total run time is 58.80 minutes.
 - Sample injection is 1µL.
- The carrier gas is helium.
- The carrier gas Flow: 6mL/min.
- Air: 400mL/min.
- Make up gas flow: 30mL/min.
- FID temperature, 310°C
- Injection port temperature, 300°C
- GC operated in split/splitless mode

7.1.2 GC Maintenance

7.1.2.1 Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert.

7.1.2.2 Break off the first few inches, up to one foot, of the injection port side of the column.

7.1.2.3 Remove the column and solvent backflush according to the manufacturer's instructions.

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7.1.2.4 Bake out the column at 300°C. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.

7.2 Retention Time Windows

- 7.2.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of the Aromatic Hydrocarbon and Aliphatic Hydrocarbon standard mixtures throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.
- 7.2.2 Calculate the standard deviation of the three absolute retention times for each individual component in the Aromatic Hydrocarbon standard, the Aliphatic Hydrocarbon standard, and all surrogate, fractionation surrogates.
- 7.2.3 Plus or minus three times the standard deviation of the absolute retention times for each standard should be used to define the retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.
- 7.2.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window or use 0.1 minutes as a default value.
- 7.2.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.
- 7.2.6 EPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of the beginning marker compound and ending 0.1 minutes after the RT of the ending marker compound, except for n-C₁₉, which is both a beginning and ending marker compound for two different ranges.

The C₉ - C₁₈ Aliphatic Hydrocarbon range ends immediately (0.1 min) before the elution of the n-C₁₉ peak. The C₁₉ - C₃₆ Aliphatic Hydrocarbon range begins 0.1 before the elution of the n-C₁₉ peak; therefore there is no overlap of the two ranges and the n-C₁₉ peak is only included in the C₁₉ - C₃₆ Aliphatic Hydrocarbon range.

EPH marker compounds and windows are summarized in the table below.

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Table - EPH Marker Compounds

Range/ Hydrocarbon Standard	Beginning Marker Cpd.	Ending Marker Compound
C ₉ -C ₁₈ Aliphatic Hydrocarbons	n-Nonane	Just Before n-Nonadecane
C ₁₉ -C ₃₆ Aliphatic Hydrocarbons	n-Nonadecane	n-Hexatriacontane
C ₁₁ -C ₂₂ Aromatic Hydrocarbons	Naphthalene	Benzo (g,h,i) Perylene

7.2.7 If a TPH analysis is done without fractionation, TPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of n-Nonane and ending 0.1 minutes after the RT of n-Hexatriacontane.

7.3 Calibration

Average calibration factors or linear regression is used to calculate the slope and y-intercept that best describes the linear relationship between EPH target analyte and range concentrations and instrument response.

Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standard solutions to volumetric flasks and diluting to volume with methylene chloride for the Aromatic standards and hexane for the Aliphatic standards. The surrogate OTP and the fractionation surrogate standards are added to the Aromatic Hydrocarbon Standard; the surrogate 5-alpha androstane is added to the Aliphatic Hydrocarbon Standard. One of the calibration standards must be at the concentration of the Reporting Limit. The other concentrations must correspond to the expected range of concentrations found in real samples or should define the working range of the detector. The following calibration levels are recommended: 1, 10, 50, 100, and 200ng/μL for the individual components. The individual and collective concentrations of standard analytes within each hydrocarbon range for these recommended calibration levels are provided in the table below.

**Table - Recommended Calibration Standard Concentrations and masses
(1μL Injection)**

Component	Conc. of std. analytes (μg/ml)				
	1	10	50	100	200
Total Mass C ₉ - C ₁₈ Aliphatic Hydrocarbons, ng (6 components)	6	60	300	600	1200
Total Mass C ₁₉ - C ₃₆ Aliphatic Hydrocarbons, ng (8 components)	8	80	400	800	1600
Total Mass C ₁₁ -C ₂₂ Aromatic Hydrocarbons/PAHs, ng (17components)	17	170	850	1700	3400

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Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak area for each compound.

7.3.1. Linear Regression

A calibration curve is prepared using area responses versus concentration. A linear calibration applying a first order equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient (r) must be greater than or equal to 0.990. The equation is:

$$y = mx + b$$

where: y = Instrument response

m = Slope of the line

x = Concentration of the calibration standard or range

b = The intercept

Calculate a linear regression (LR) for the individual PAH compounds that comprise the Aromatic Hydrocarbon standard. This is not necessary if the Target or Diesel PAH Analytes will not be individually identified and quantitated using the EPH method.

Calculate a LR for the extraction and fractionation surrogates.

A collective calibration curve must also be established for each hydrocarbon range of interest using the FID chromatogram of the appropriate fraction: C₉-C₁₈ Aliphatic Hydrocarbons, C₁₉-C₃₆ Aliphatic Hydrocarbons, and C₁₁-C₂₂ Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. A listing of the collective concentrations of standards within each hydrocarbon range is provided in the table in section 7.3.

Note: The area for the surrogates must be subtracted from the area summation of the range in which they elute (e.g., 5-alpha androstane is subtracted from the C₁₉ - C₃₆ Aliphatic Hydrocarbon range). Do not include the area of naphthalene or 2-methylnaphthalene in the linear regression analysis of the C₉-C₁₈ Aliphatic Hydrocarbon range.

7.3.2 The average calibration factor procedure

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The ratio of area response to the concentration injected, defined as the calibration factor (CF), may be calculated for target PAH compounds using the equation below.

Calibration Factor (CF) = area of peak/concentration injected (ug/L)

The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest as determined using the equation below. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.

$\%RSD = (\text{stand dev of 5 CFs} / \text{mean of 5 CFs}) \times 100$

A collective calibration factor must also be established for each hydrocarbon range of interest using the FID chromatogram of the appropriate fraction: C₉-C₁₈ Aliphatic Hydrocarbons, C₁₉-C₃₆ Aliphatic Hydrocarbons, and C₁₁-C₂₂ Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The results can be used to calculate the ratio of the peak area response summation to the concentration injected, defined as the CF, for the hydrocarbon ranges using the equation below. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest. A listing of the collective concentrations of standards within each hydrocarbon range is provided in the table in section 7.3.

Note: The area for the surrogates must be subtracted from the area summation of the range in which they elute (e.g., 5-alpha androstane is subtracted from the C₁₉ - C₃₆ Aliphatic Hydrocarbon range). Do not include the area of naphthalene or 2-methylnaphthalene in the analysis of the C₉-C₁₈ Aliphatic Hydrocarbon range.

- 7.3.3. For TPH analyses, without fractionation, calculate a collective calibration curve. Tabulate the summation of the peak areas of all component standards in the aliphatic fraction (i.e., 14 components) against the total mass injected. Do not include any surrogates.
- 7.3.4 At a minimum, the working calibration curve must be verified on each working day, after every 20 samples or 24 hours (whichever is more frequent), and at the end of the sequence, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than $\pm 25\%$ ($\pm 30\%$ for n-nonane), as calculated using the equation below, instrument

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below, instrument maintenance must be performed (such as changing the liner) and/or a new calibration curve must be determined for that analyte or range.

Linear Calibration Percent Difference (%D)

$$\%D = (R_1 / R_2) \times 100$$

where: R1 = Calculated concentration from curve.
R2 = Expected concentration.

For the closing continuing calibration standard, four compounds may exhibit percent differences or percent drifts greater than 25 % but less than 40 %.

Average Calibration Factor Percent Difference (%D)

$$\%D = (CF_{avg} - CF_{cc}) / (CF_{avg}) \times 100$$

where: CF_{avg} = Average calibration factor calculated from initial calibration
CF_{cc} = Calibration factor calculated from continuing calibration

7.4 GC Analysis

7.4.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (or calibration verification) followed by sample extracts interspersed with blanks and QC samples, and closes with a mid-range continuing calibration verification. The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.4.2 Aliphatic and aromatic extracts are introduced into the gas chromatograph by direct injection of 1 µl of sample.

7.4.3 Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.2. Alternately, the default value of 0.1 minutes may be used for the daily retention time window.

7.4.3.1 Tentative identification of an aromatic analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation is performed by reanalysis on a dissimilar GC column.

7.4.3.2 Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the

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analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

- 7.4.4 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peak eluted between specified range "marker" compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual check be initially performed, to document proper integration functions.
- 7.4.5 When quantifying on a peak area basis by internal or external calibration, collective peak area integration for the fractional ranges, or TPH, must be from baseline (i.e. must include the unresolved complex mixture "hump" areas). For the integration of individual Target Analytes, surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.
- 7.4.6 Baseline correction using an instrument thermal bleed is permissible, if conducted in accordance with the procedures and requirements specified in Section 7.6.5.
- 7.4.7 If the concentration of a Target or Diesel PAH Analyte, the aliphatic range C₉ through C₁₈, aliphatic range C₁₉ through C₃₆, or aromatic range C₁₁ through C₂₂ exceed(s) the calibration range of the curve, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale, and bracketed by upper and lower calibration standards. Overlapping peaks are not always evident when peaks are off scale.
- 7.4.8 For non-target peaks eluting in the aliphatic, aromatic or TPH fractions, the upper linear range of the system should be defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range for the detector. If any non-target peak eluting within any aliphatic or aromatic range exceeds twice the peak height documented for the highest range-specific calibration standard, dilute the extract and reanalyze.

7.5 Calculations

7.5.1 Linear Regression Analysis

The concentration of each analyte and/or hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, using linear regression analysis.

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7.5.1.1. The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

$$\text{Amt} = (y - b) / m$$

Where: Amt = Concentration calculated by Target in ug/mL.
y = Instrument response
m = Slope of the line
b = The intercept

7.5.2 Average Calibration Factor Analysis

The concentration of each analyte and/or hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, using CF's.

7.5.1.2. The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibration method.

$$\text{Amt} = x/m$$

Where: Amt = Concentration calculated by Target in ug/mL.
x = Instrument response
M = average CF

7.5.3 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = Amt x DF [(Vt / Vo) x 1000]

Soil: Concentration (mg/Kgdrywt) = Amt x DF [(Vt / Vo) x (100/(100-M))]

Where: DF = Dilution factor.
Vt = Final extract volume in L.
Vo = Sample volume in L or kg.
M = % Moisture.

7.6 Sample Analysis

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7.6.1 Aliphatic Fraction

7.6.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C₉ and 0.1 minutes before the Rt for n-C₁₉. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.1.2 Determine the total area count for all peaks eluting 0.1 minutes before the Rt for n-C₁₉ and 0.1 minutes after the Rt for n-C₃₆. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.1.3 Determine the peak area count for the sample surrogate standard (5-alpha androstane) and any internal standard used. Subtract these values from the collective area count value within the appropriate hydrocarbon range(s).

7.6.1.4 Using linear regression in Target, calculate the collective concentrations of C₉ through C₁₈ Aliphatic Hydrocarbons, C₁₉ through C₃₆ Aliphatic Hydrocarbons, and individual concentrations of any sample surrogate.

7.6.2 Aromatic Fraction

7.6.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.

7.6.2.2 Determine the peak area count for the sample surrogate (OTP), the fractionation surrogates, and any internal standard used. Subtract these values from the collective area count.

7.6.2.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

7.6.2.4 Using linear regression in Target, calculate the concentrations of Unadjusted C₁₁ through C₂₂ Aromatic Hydrocarbons, the sample surrogate standard (OTP), the fractionation surrogates, and, optionally, the Target or Diesel PAH Analytes.

7.6.2.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the

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concentration of Unadjusted C₁₁ - C₂₂ Aromatic Hydrocarbons. If the concentration of Target or Diesel PAH Analytes were not determined, report a value for unadjusted C₁₁ - C₂₂ Aromatics, and indicate "Not Determined" for C₁₁ - C₂₂ Aromatics.

7.6.3 Total Petroleum Hydrocarbons

7.6.3.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for n-C₉ and 0.1 minutes after the RT for n-C₃₆. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

7.6.3.2 Determine the peak area count for any surrogate. Subtract these values from the collective area count value.

7.6.3.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

7.6.3.4 Using linear regression in Target, calculate the concentration of Unadjusted TPH, and, optionally, the Target or Diesel PAH Analytes.

7.6.3.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the concentration of unadjusted TPH. If the concentration of Target or Diesel PAH Analytes were not determined, report a value for Unadjusted TPH, and indicate "Not Determined" for TPH.

7.6.4 Data Manipulations

7.6.4.1 By definition, the collective concentration of the aromatic fraction (and/or TPH) **excludes** the individual concentrations of the Target PAH Analytes. Accordingly, a data manipulation step is performed in KIMS that subtracts the individual PAH analyte concentrations.

7.6.4.2 Subtract the individual concentrations of the Target or Diesel PAH Analytes from the collective concentration of Unadjusted C₁₁ through C₂₂ Aromatic Hydrocarbons and/or Unadjusted TPH. If the individual concentrations of Target Analytes have not been quantitated, report a value for Unadjusted C₁₁ through C₂₂ Aromatic Hydrocarbons and/or Unadjusted TPH, and indicate "Not Determined" for C₁₁ through C₂₂ Aromatic Hydrocarbons and/or TPH.

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7.6.5 Baseline Correction for Instrument Noise Level

7.6.5.1 Range integration areas are corrected by the automatic subtraction of the baseline established by activation of an instrument thermal bleed.

7.6.5.2 If baseline correction is used, instrument baseline must be established for every batch of samples, and after the analysis of samples that are suspected to be highly contaminated.

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS/LCSD, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the method acceptance limits of 40-140%. The sample is evaluated for recoveries of the surrogates. For the aliphatic extract, if the recovery of 5-alpha androstane is high and the sample results are less than the PQL, narrate. If the recovery is low and may be attributable to matrix interference, reanalyze to confirm a matrix effect and narrate. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract.

For the aromatic fraction, if the recovery of OTP is low and the fractionation surrogates are low, re-fractionate. If the fractionation surrogates are acceptable and the OTP is low and may be attributable to matrix interference,

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interference, reanalyze to confirm a matrix effect and narrate. If the OTP is low and is not attributable to matrix interference, the sample results are less than the PQL, and the fractionation surrogates are acceptable, re-extract.

7.7.3 Chromatography

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Any manual integrations that are necessary (for instance, if the sample contains a concentration of TPH/DRO which was integrated “valley to valley” instead of a “baseline to baseline”), are performed in Target Review. An “m” qualifier will automatically be printed on the quantitation report summary. The analyst must also date and initial in the space next to each of these qualifiers. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

7.7.4 Target Compound Detection

The aromatic analysis chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within $\pm 50\%$, the concentration from channel A is reported.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the RL (Reporting Limit – based on the lowest calibration standard), if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ drastically, or if an analyte is present but its retention time is outside of the retention time window for that analyte.

7.8 Reporting

7.8.1 After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, reports, such as chronology or calibration forms, are generated. Reports of Analysis

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(ROA), LCS/LCSD, MS/MSD and surrogate forms are generated in KIMS. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

- 7.8.2 The EPH reporting form contains an attestation, which indicates whether significant modifications were made to the EPH method, a clear affirmation on whether the QA/QC procedures and standards specified in the method were followed and achieved.

Significant modifications may include a different fractionation procedure than the one specified in the method, a change in the extraction procedure, the use of different surrogates. Any modifications, which were made along with any QA/QC deviations, are mentioned in the case narrative.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 4 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 4, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 4 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 4 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

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8.1 General Requirements and Recommendations

- 8.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated.
- 8.1.2 A system solvent blank must be run after a sample suspected of being highly contaminated to determine if sample carryover has occurred.
- 8.1.3 At a minimum, for each analytical batch (up to 20 samples), a beginning and ending Calibration Check Standard, Method Blank, Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) must be run, and a Matrix Sample (MS) and/or MS duplicate or sample duplicate (DUP) should be analyzed, at the discretion of the analyst, based upon the nature of the sample. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered. The blank and spiked samples should be carried through all stages of the sample preparation and measurement process.
- 8.1.4 The recommended sequence of analysis is as follows:
- Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration) [REQUIRED]
 - Method Blank [REQUIRED]
 - Laboratory Control Sample and Laboratory Control Sample Duplicate [REQUIRED]
 - Samples [up to 20]
 - Matrix Sample/duplicate [As appropriate/Client Requested]
 - Mid-range Calibration Check Standard [consider after 10 samples, as appropriate] [REQUIRED after 20 samples or at end of analytical sequence]
- 8.1.5 At a minimum, when the surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, reanalyze the sample.
- 8.1.6 Each sample and QC sample must be evaluated for potential breakthrough on a sample-specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic

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fractions of the LCS and LCSD. If the concentration of either naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration (sum of analyte in aromatic and aliphatic fraction) for naphthalene and 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all archived batch extracts. If the fractionation surrogate recovery is outside the 40-140% limits, then fractionation must be repeated on the archived extract of the affected samples.

8.2 Minimum Instrument QC

8.2.1 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

8.2.1.1 The n-nonane (n-C₉) peak must be adequately resolved from the solvent front of the chromatographic run.

8.2.1.2 The surrogates COD and 5-alpha androstane, fractionation surrogate standards must be adequately resolved from any individual components in the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.

8.2.1.3 All peaks of interest from the Aliphatic Hydrocarbon standard must be adequately resolved to baseline. In the Aromatic Hydrocarbon standard, baseline separation is expected for Phenanthrene and Anthracene. Benzo(a)Anthracene, Chrysene, Benzo(b)Fluoranthene, Benzo(k)fluoranthene, Dibenzo(a,h)Anthracene, and Indeno(1,2,3-cd)Pyrene are not expected to be chromatographically separated to baseline; however, sufficient separation should be obtained to 50% baseline.

8.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.

8.2.3 Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels and the correlation coefficient (r) must be at least 0.990.

8.2.4 The calibration ranges of the aliphatic and aromatic hydrocarbon fractions, and/or TPH fraction, are based on the respective high concentration standard.

8.2.5 In order to demonstrate the absence of mass discrimination, the response ratio of C₂₈ to C₂₀ must be at least 0.85. If <0.85, this nonconformance must

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be noted in the laboratory case narrative. The chromatograms of Continuing Calibration Standards for aromatics must also be reviewed to ensure that there are no obvious signs of mass discrimination.

8.2.6 Due care must be exercised to assure that the peaks for naphthalene and n-dodecane in the aliphatic hydrocarbon fraction are resolved to allow for an accurate determination of the naphthalene concentration in the LCS/LCSD pair.

8.3 Initial and Periodic Method QC Demonstrations

The procedures specified in Section 8.3.1 through 8.3.3 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

8.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate laboratory reagent grade water and/or clean sand blanks spiked with each analyte of interest at approximately 50µg/L and/or 5mg/kg, respectively.

8.3.1.1 Extract each replicate according to the procedures described in the current revision of Katahdin SOP CA-511. Analyze according to the procedures described in section 7.0.

8.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

8.3.1.3 For each analyte, excluding n-C₃₆, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%. Poorer recoveries may be experienced for the n-C₃₆ standard. For each analyte, the %RSD must be less than or equal to 25% (30% for n-nonane).

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8.3.2 Fractionation

8.3.2.1 To demonstrate the capability of properly fractionating aliphatic and aromatic hydrocarbons in a sample, the analyst must first prepare and analyze the Fractionation Check Solution specified in Section 5.6, using the HPLC fractionation procedure.

8.3.2.2 For each analyte within the Fractionation Check Solution, excluding n-C₃₆, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%.

8.4 Ongoing Method QC Demonstrations

8.4.1 Each sample, blank, and LCS/LCSD must be spiked with the surrogate spiking solution. Required surrogate recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

8.4.2 Each sample extract must be spiked with a fractionation surrogate spiking solution prior to fractionation. Required recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

8.4.3 At a minimum, with every batch of 20 samples or less the lab must analyze the following:

8.4.3.1 Independent Calibration Verification (ICV) Standard - A mid-range calibration standard, prepared from a source independent from the stock standard solution used to develop the calibration curve. This standard must be analyzed prior to sample analysis to verify the calibration. If the percent difference (%D) of any analyte, within a calibration check standard, varies from the predicted response by more than 25% (30% for n-nonane), corrective action must be performed (such as changing the reprep of the standard) and/or a new calibration curve must be prepared

8.4.3.2 Calibration Check Standard - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to and after sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered after the analysis of the tenth sample. If the percent difference (%D) of any analyte, within a calibration check standard,

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varies from the predicted response by more than 25% (30% for n-nonane), instrument maintenance must be performed (such as changing the liner) and/or a new calibration curve must be prepared for that analyte.

8.4.3.3 Method Blank - A water or soil Method Blank is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the surrogate spiking solution. Peaks detected within the retention time window of any analyte or range of interest above a Reporting Limit must be noted on the data report form.

8.4.3.4 Laboratory Control Sample - A Laboratory Control Sample is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the matrix spiking solution. The spike recovery should be between 40% and 140%, excluding n-C₃₆.

8.4.3.5 Laboratory Control Sample Duplicate - A Laboratory Control Sample duplicate is prepared by fortifying a laboratory reagent grade water or clean sand blank with 1.0mL of the matrix spiking solution. The spike recovery should be between 40% and 140%, excluding n-C₃₆. The RPD between the laboratory control sample and laboratory control sample duplicate should be less than 25%.

8.4.3.6 Instrument Thermal Bleed - If baseline correction will be employed, a instrument thermal bleed must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated.

8.4.4 At the discretion of the analyst, and in consideration of sample matrices and data quality objectives, it is recommended that with every batch of 20 samples or less the lab consider analysis of the following:

8.4.4.1 Matrix Sample (MS)/Duplicate (MSD) - The water or soil MS is prepared by fortifying an actual water or soil sample with 1.0mL of the matrix spiking solution. The purpose of the MS spike is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations. The corrected concentrations of each analyte within the MS spike sample should be within 40 to 140% of the true value, excluding n-C₃₆. The %RPD of MS Duplicates should be less than 50%.

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- 8.4.5 If any of the performance standards specified in Section 8.4.1 through 8.4.3 are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are fallen out must be rerun. If this is not possible, that data must be reported as suspect.
- 8.4.6 The analyte and hydrocarbon range reporting limits should be verified/re-established at least once per year, or upon a major change in system equipment or operations.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Inorganic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", for procedures on determining the MDL.

The Practical Quantitation Limit (PQL) concentrations for all the target analytes are listed in Table 5.

Refer to the current revision Method MADEP EPH-04-1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, May 2004 revision 1.1

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, "Equipment Maintenance and Troubleshooting", current revision.

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.

Katahdin Analytical Services, Inc. SOP CA-511, "Extraction of Petroleum Hydrocarbons from Samples for Analysis by MADEP - EPH Methods", current revision

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**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP EPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five-point external calibration of 17 targeted PAH standards, and collective calibrations of C ₁₁ through C ₂₂ aromatic hydrocarbons, C ₉ through C ₁₈ aliphatic hydrocarbons and C ₁₉ through C ₃₆ aliphatic hydrocarbons.	Initial calibration prior to sample analysis	The correlation coefficient (r) must be greater than or equal to 0.99. CF must be equal to or less than 25%	Investigate and repeat initial calibration
Initial Calibration Verification (ICV)	Once after each calibration	All analytes ≤ 25 %D of the expected value.	Reanalyze sample Reprepare standard Reprepare standard from fresh stock.
CV	If initial calibration analyzed, daily and after 10 to 20 samples, and at end of sequence.	%RPD within 30% for n-nonane and for all other analytes within 25% The closing CCV may have up to 4 compounds > 25% but less than 40% D.	Evaluate the samples: If the %RPD >25% (30% for n-nonane) and sample results are < PQL, narrate. If %RPD >25% (30% for n-nonane) and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. if the blank results are above the PQL, report samples results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS/LCSD	One LCS/LCSD pair per prep batch	Spike recovery must be between 40% and 140% and RPD less than 25%	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
LCS/LCSD	One LCS/LCSD pair per prep batch	Concentration of naphthalene and 2-methylnaphthalene in the aliphatic fraction must be less than 5% of total concentration	Refractionate all archived extracts from the extraction batch

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 1, cont'd

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD MADEP EPH

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate	Every sample, blank, and QC sample	Recovery must be between 40% and 140%	Refer to section 7.7.2
Matrix Spike/Matrix Spike Duplicate	One MS/MSD per batch of 20 samples (optional), as requested by clients.	Recovery must be between 40% and 140% and RPD less than 50%	(1) Evaluate the samples and associated QC: i.e. If the LCS is acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample duplicate if requested by the client	One per batch of 20 samples	%RPD of duplicate must be less than 50%.	(1) check calculations for errors (2) Evaluate QC
Fractionation Check Solution	Prepare for each new lot of silica gel cartridge lot or every 3 months, whichever is sooner	Mean accuracy must be between 40 and 140%.	Investigate and refractionate.
Demonstration of capability - four replicate analyses of a QC check sample	One time per analyst initially and annually thereafter	For each analyte, the mean accuracy must be 40 to 140 %R and the %RSD must be < 25%	Investigate; reprep
MDL and/or LOD/LOQ Verification	Refer to the current revision of Katahdin SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", for procedures on determining the MDL.		

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-322-08	METHOD MADEP - EPH
Apparatus/ Materials		
Reagents/ Standards	1) Aliphatic hydrocarbon standards prepared in methylene chloride.	1) Aliphatic hydrocarbon standards prepared in hexane.
Sample preservation/ handling	1) Soil samples collected in clear jars	1) Soil samples collected in amber jars
Procedures	1) Baseline correction made by the automatic subtraction of the baseline established by activation of a instrument thermal bleed 2) Prepare petroleum reference spiking solution by weighing 0.0100g of neat product	1) Baseline correction made by the subtraction of the baseline established by the injection of a system solvent blank 2) Prepare petroleum reference spiking solution by weighing 0.0250g of neat product
QC - Standards	1) Surrogate, spike and fractionation surrogate all prepared at 90 ug/mL for use in a 2 mL or prefractionated volume. 2) Fractionation check solution: aliphatics, aromatics, 5-alpha androstane, and o-terphenyl at 200ug/mL each	1) Surrogate at 40 ug/mL, matrix spike at 50-150 ug/mL, fractionation surrogate at 40 ug/mL. 2) Fractionation check solution: aliphatics and aromatics at 200ug/mL each
QC - LCS		
QC - Accuracy/ Precision		
QC - MDL		

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 3

HOLDING TIMES AND PRESERVATIVES FOR EPH SAMPLES

MATRIX	CONTAINER	PRESERVATION	HOLDING TIME
Aqueous Samples	1 Liter amber glass bottle with Teflon-lined screw cap	Add 5mL of 1:1HCl; cool to 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120mL) Wide-mouth glass jar with Teflon-lined screw cap	Cool to 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth glass jar with Teflon-lined screw cap. Jar should be filled to only 2/3 capacity to avoid breakage if expansion occurs during freezing.	Freeze at -10 °C in the field or in the laboratory*	Samples must be extracted within 14 days of the date thawed and extracts analyzed within 40 days of extraction*

*Samples processed in the laboratory must be preserved at 4 (±2) °C and frozen within 48 hours of the time of collection. Frozen samples may be held for up to one year prior to analysis and must be extracted within 24 hours of thawing.

TABLE 4

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, MATRIX SPIKE AND
CALIBRATION STANDARD CONCENTRATIONS - MADEP - EPH

ANALYTE	Stock *	Fractionation *	Matrix Spike	Calibration Standard Concentrations (in MeCl ₂)				
	Standards	Check Solution	Standard ***	(ng/μL)	(ng/μL)	(ng/μL)	(ng/μL)	(ng/μL)
	(ng/μL)	(ng/μL)	(ng/μL)	level 1	level 2	level 3	level 4	level 5
Naphthalene	1,000	200	90	1	10	50	100	200
2-Methylnaphthalene	1,000	200	90	1	10	50	100	200
Acenaphthylene	1,000	200	90	1	10	50	100	200
Acenaphthene	1,000	200	90	1	10	50	100	200
Fluorene	1,000	200	90	1	10	50	100	200
Phenanthrene	1,000	200	90	1	10	50	100	200
Anthracene	1,000	200	90	1	10	50	100	200
Fluoranthene	1,000	200	90	1	10	50	100	200
Pyrene	1,000	200	90	1	10	50	100	200
Benzo(a)Anthracene	1,000	200	90	1	10	50	100	200
Chrysene	1,000	200	90	1	10	50	100	200
Benzo(b)Fluoranthene	1,000	200	90	1	10	50	100	200
Benzo(k)Fluoranthene	1,000	200	90	1	10	50	100	200
Benzo(a)Pyrene	1,000	200	90	1	10	50	100	200
Indeno(1,2,3-cd)Pyrene	1,000	200	90	1	10	50	100	200
Dibenzo(a,h)Anthracene	1,000	200	90	1	10	50	100	200
Benzo(g,h,i)Perylene	1,000	200	90	1	10	50	100	200
Ortho-Terphenyl (surr)	1,000	200	----	1	10	50	100	200
Nonane	1,000	200	90	1	10	50	100	200
Decane	1,000	200	90	1	10	50	100	200
Dodecane	1,000	200	90	1	10	50	100	200
Tetradecane	1,000	200	90	1	10	50	100	200
Hexadecane	1,000	200	90	1	10	50	100	200
Octadecane	1,000	200	90	1	10	50	100	200
Nonadecane	1,000	200	90	1	10	50	100	200
Eicosane	1,000	200	90	1	10	50	100	200
Docosane	1,000	200	90	1	10	50	100	200
Tetracosane	1,000	200	90	1	10	50	100	200
Hexacosane	1,000	200	90	1	10	50	100	200
Octacosane	1,000	200	90	1	10	50	100	200
Triacontane	1,000	200	90	1	10	50	100	200
Hexatriacontane	1,000	200	90	1	10	50	100	200
5-alpha androstane(surrogate)	1,000	200	-----	1	10	50	100	200

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 4, cont'd

RECOMMENDED STOCK, FRACTIONATION CHECK SOLUTION, MATRIX SPIKE AND
CALIBRATION STANDARD CONCENTRATIONS - MADEP - EPH

- * The Aromatic Hydrocarbon Standards (17 PAH compounds and ortho-Terphenyl) should be prepared in methylene chloride. The Aliphatic Hydrocarbon Standards (consisting of 14 normal alkanes and 1-chlorooctadecane) should be prepared in Methylene chloride.
- ** The Fractionation Check Standard should be prepared in hexane.
- *** The Matrix Spike Solution should be prepared in acetone.

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

TABLE 5

PQLs FOR METHOD MADEP EPH

PARAMETER/ METHOD	ANALYTE	PRACTICAL QUANTITATION LEVEL (PQL)		
		Aqueous (µg/L)	Soil (mg/kg)	
Extractable Petroleum Hydrocarbons	Naphthalene	2	0.2	
	2-Methylnaphthalene	2	0.2	
	Acenaphthylene	2	0.2	
	Acenaphthene	2	0.2	
	Fluorene	2	0.2	
	Phenanthrene	2	0.2	
	Anthracene	2	0.2	
	Fluoranthene	2	0.2	
	Pyrene	2	0.2	
	Benzo(a)Anthracene	2	0.2	
	Chrysene	2	0.2	
	Benzo(b)Fluoranthene	2	0.2	
	Benzo(k)Fluoranthene	2	0.2	
	Benzo(a)Pyrene	2	0.2	
	Indeno(1,2,3-cd)Pyrene	2	0.2	
	Dibenzo(a,h)Anthracene	2	0.2	
	Benzo(g,h,i)Perylene	2	0.2	
			2	0.2
		C ₉ - C ₁₈ Aliphatics	100	20
		C ₁₉ - C ₃₆ Aliphatics	100	20
	C ₁₁ - C ₂₂ Aromatics	100	20	

TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD

FIGURE 1

EXAMPLE OF MADEP-EPH ANALYTICAL LOGBOOK PAGE

Katahdin Analytical Services, Inc. GC Laboratory Instrument Runlog
Instrument: GC12 (FID) Amount Injected 1ul Method (circle) EPH(MADEP) / FL PRO / TNRC 1005
Reviewed by/ Date: _____ DRO/TPH - 8015Mod. / MDEP 4.1.25 / 8100Mod.

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
4-2-10	JLP	00010 37	GCO7361	Y	AR0029A	334	SECS JLP
4-3-10		38	SD1425-5 3910				OTPL
		39	-6				AN SS ↓
		40	-7				OTPL
		41	-8				
		42	-9				
		43	↓ -10 ↓				OTPL
		44	SD1390-10				PF
		45	MeClz	N			
		46	TB	Y			
		47	ARO Sample	Y			↑ H1923
		48	TB	N			
		49	↓				
		50	↓				
4-5-10	JLP	51	TB	N			
		52	TB	Y			
		53	ARO Sample	Y			H1923
		54	WG75480-1 3510	Y			
		55	↓ -2	Y			
		56	↓ -3				
		57	SD1447-62				
		58	↓ -3				
		59	-4				
		60	-5				
		61	↓ -6				
		62	TB	Y			
		63	ARO Sample	Y			H1923
		64	TB	N			
		65	↓				
4-6-10	JLP	66	TB				

**TITLE: METHOD FOR THE ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS
MADEP - EPH METHOD**

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ (Verbals turned in). Due Date _____

Client:	Primary	Secondary
Method:	Date:	Date:
SDG No: Level:	Initials:	Initials:
KAS No:	Approved : <input type="checkbox"/> Yes	

DODQSM 3.0 **DODQSM 4.1** **WITH LAB. LIMITS**
QUAPP **LAB** (*REPORT ND's to MDL*)

List all curves that are scanned. _____

Narrate which QC limits were used for (Surr., LCS,s MS/MSDs.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria. _____

Narrate any method deviations. (Blanks, LCS,s etc.) _____

Sign & Date Manual integration (**Narrate as needed**). _____

Sample I.D's Truncated (**NARRATED**). YES Please list KAS # below :

Enco SOPs and Accreditations



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

ENVIRONMENTAL CONSERVATION LABORATORIES – ORLANDO
 10775 Central Port Drive
 Orlando, FL 32824
 Dorian Pearson-Shaver Phone: 407 826 5314
 dpearsonshaver@encolabs.com

ENVIRONMENTAL

Valid To: March 31, 2012

Certificate Number: 3000.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, 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)) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
<u>Metals</u>	EPA 6020A/200.8	EPA 6020A
Aluminum	EPA 6020A/200.8	EPA 6020A
Antimony	EPA 6020A/200.8	EPA 6020A
Arsenic	EPA 6020A/200.8	EPA 6020A
Barium	EPA 6020A/200.8	EPA 6020A
Beryllium	EPA 6020A/200.8	EPA 6020A
Cadmium	EPA 6020A/200.8	EPA 6020A
Calcium	EPA 6020A/200.8	EPA 6020A
Chromium	EPA 6020A/200.8	EPA 6020A
Cobalt	EPA 6020A/200.8	EPA 6020A
Copper	EPA 6020A/200.8	EPA 6020A
Hardness	SM 2340 B	-----
Iron	EPA 6020A/200.8	EPA 6020A
Lead	EPA 6020A/200.8	EPA 6020A
Magnesium	EPA 6020A/200.8	EPA 6020A
Manganese	EPA 6020A/200.8	EPA 6020A
Mercury	EPA 245.1/7470A	EPA 7471B
Molybdenum	EPA 6020A/200.8	EPA 6020A
Nickel	EPA 6020A/200.8	EPA 6020A
Potassium	EPA 6020A/200.8	EPA 6020A
Selenium	EPA 6020A/200.8	EPA 6020A
Silver	EPA 6020A/200.8	EPA 6020A

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
Sodium	EPA 6020A/200.8	EPA 6020A
Thallium	EPA 6020A/200.8	EPA 6020A
Tin	EPA 6020A/200.8	EPA 6020A
Titanium	EPA 6020A/200.8	EPA 6020A
Vanadium	EPA 6020A/200.8	EPA 6020A
Zinc	EPA 6020A/200.8	EPA 6020A
<u>Microbiology</u>		
Total Coliforms	SM 9222B	-----
Fecal Coliforms	SM 9222D	-----
<u>General Chemistry</u>		
Acidity, as CaCO ₃	EPA 305.1/SM 2310 B (4A)	-----
Alkalinity as CaCO ₃	EPA 310.1/SM 2320 B	EPA 310.1/SM 2320 B
Alkalinity as CaCO ₄	EPA 310.2	EPA 310.2
Ammonia as N	-----	EPA 350.1
Biochemical oxygen demand	EPA 405.1/SM 5210 B	-----
Bromide	EPA 300.0/9056A	EPA 9056A
Carbonaceous BOD (CBOD)	SM 5210 B	-----
Chemical oxygen demand	EPA 410.4	-----
Chloride	EPA 300.0/9056A	EPA 9056A
Chromium VI	EPA 7196/ SM 3500-Cr D	EPA 7196
Conductivity	EPA 120.1	-----
Cyanide	EPA 335.2/SM 4500-CN E	EPA 9014
Ferric iron (calculated)	SM 3500-Fe D	-----
Ferrous iron	SM 3500-Fe D	-----
Fluoride	EPA 300.0/9056A	EPA 9056A
Hardness	EPA 130.2/SM 2340 C	-----
Kjeldahl nitrogen -total	EPA 351.2	EPA351.2
Nitrate as N	EPA 300.0/353.1/9056A	EPA 353.1/9056A
Nitrate-nitrite	EPA 300.0/353.1/9056A	EPA 353.1/9056A
Nitrite as N	EPA 300.0/354.1/9056A/SM 4500-NO ₂ B	EPA 9056A/ SM 4500-NO ₂ B
Organic nitrogen	EPA 351.2/350.1	EPA 351.2/350.1
Orthophosphate as P	EPA 365.1	-----
Orthophosphate as P	EPA 365.3	-----
pH	EPA 150.1/9040C/SM 4500-H ⁺ -B	EPA 9040C
Phosphorus, total	EPA 365.4	EPA 365.4
Residue-filterable (TDS)	SM 2540 C	-----
Residue-nonfilterable (TSS)	SM 2540 D	-----
Residue-total	SM 2540 B/SM 2540 G/EPA 160.3	SM 2540G/EPA 160.3
Residue-volatile	EPA 160.4	EPA 160.4
Sulfate	EPA 300.0/9056A	EPA 9056A
Sulfide	EPA 376.1/SM 4500-S E	-----
Surfactants -MBAS	SM 5540 C	-----
Total nitrate-nitrite	EPA 9056 A/SM 4500-NO ₃ H	EPA 9056 A/SM 4500-NO ₃ H
Total cyanide	EPA 9014	EPA 9014
Total nitrogen	TKN + Total nitrate-nitrite	TKN + Total nitrate-nitrite
Total Organic Carbon	EPA 9060A/SM 5310B	TOC Walkley Black
Total phenolics	EPA 420.1	EPA 420.1
Total, fixed, and volatile residue	SM 2540 G	SM 2540 G
Turbidity	EPA 180.1	-----

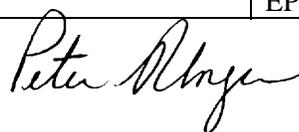
Peter Abney

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
Un-ionized ammonia	DEP SOP 10/03/83	DEP SOP 10/03/83
<u>Extractable Organics</u>		
1,2,4-Trichlorobenzene	EPA 8270D/625	EPA 8270D
1,2,4,5-Tetrachlorobenzene	EPA 8270D/625	EPA 8270D
1,2-Dichlorobenzene	EPA 8270D/625	EPA 8270D
1,3-Dichlorobenzene	EPA 8270D/625	EPA 8270D
1,4-Dichlorobenzene	EPA 8270D/625	EPA 8270D
1-Methylnaphthalene	EPA 8270D/625	EPA 8270D
2,3,4,6-Tetrachlorophenol	EPA 8270D/625	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D/625	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D/625	EPA 8270D
2,4-Dichlorophenol	EPA 8270D/625	EPA 8270D
2,4-Dimethylphenol	EPA 8270D/625	EPA 8270D
2,4-Dinitrophenol	EPA 8270D/625	EPA 8270D
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270D/625/ Scan-Sim	EPA 8270D
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270D/625	EPA 8270D
2-Chloronaphthalene	EPA 8270D/625	EPA 8270D
2-Chlorophenol	EPA 8270D/625	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 8270D/625	EPA 8270D
2-Methylnaphthalene	EPA 8270D/625	EPA 8270D
2-Methylphenol (o-Cresol)	EPA 8270D/625	EPA 8270D
2-Nitroaniline	EPA 8270D/625	EPA 8270D
2-Nitrophenol	EPA 8270D/625	EPA 8270D
3,3'-Dichlorobenzidine	EPA 8270D/625	EPA 8270D
3/4-Methylphenols (m/p-Cresols)	EPA 8270D/625	EPA 8270D
3-Nitroaniline	EPA 8270D/625	EPA 8270D
4-Bromophenyl phenyl ether	EPA 8270D/625	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D/625	EPA 8270D
4-Chloroaniline	EPA 8270D/625	EPA 8270D
4-Chlorophenyl phenyl ether	EPA 8270D/625	EPA 8270D
4-Nitrophenol	EPA 8270D/625	EPA 8270D
Acenaphthene	EPA 8270D/625	EPA 8270D
Acenaphthylene	EPA 8270D/625	EPA 8270D
4-Methylphenol (p-Cresol)	EPA 8270D/625	EPA 8270D
4-Nitroaniline	EPA 8270D/625	EPA 8270D
Acetophenone	EPA 8270D/625	EPA 8270D
Anthracene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Atrazine	EPA 8270D/625	EPA 8270D
Benzaldehyde	EPA 8270D/625	EPA 8270D
Benzidine	EPA 8270D/625/ Scan-Sim	EPA 8270D
Benzo(a)anthracene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Benzo(a)pyrene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Benzo(g,h,i)perylene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D/625/ Scan-Sim	EPA 8270D
1,1-Biphenyl	EPA 8270D/625	EPA 8270D
bis(2-Chloroethoxy) methane	EPA 8270D/625	EPA 8270D
bis(2-Chloroethyl) ether	EPA 8270D/625	EPA 8270D
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270D/625	EPA 8270D

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270D/625	EPA 8270D
Butyl benzyl phthalate	EPA 8270D/625	EPA 8270D
Caprolactam	EPA 8270D/625	EPA 8270D
Carbazole	EPA 8270D/625	EPA 8270D
Chrysene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Dibenz(a,h)anthracene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Dibenzofuran	EPA 8270D/625	EPA 8270D
Diethyl phthalate	EPA 8270D/625	EPA 8270D
Dimethyl phthalate	EPA 8270D/625/ Scan-Sim	EPA 8270D
Di-n-butyl phthalate	EPA 8270D/625	EPA 8270D
Di-n-octyl phthalate	EPA 8270D/625	EPA 8270D
Fluoranthene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Fluorene	EPA 8270D/625	EPA 8270D
Hexachlorobenzene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Hexachlorobutadiene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D/625	EPA 8270D
Hexachloroethane	EPA 8270D/625	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D/625/ Scan-Sim	EPA 8270D
Isodrin	EPA 8270D/625	EPA 8270D
Isophorone	EPA 8270D/625	EPA 8270D
Naphthalene	EPA 8270D/625	EPA 8270D
Nitrobenzene	EPA 8270D/625	EPA 8270D
n-Nitrosodi-n-propylamine	EPA 8270D/625	EPA 8270D
n-Nitrosodiphenylamine	EPA 8270D/625	EPA 8270D
Pentachlorophenol	EPA 8270D/625/ Scan-Sim	EPA 8270D
Phenanthrene	EPA 8270D/625	EPA 8270D
Phenol	EPA 8270D/625	EPA 8270D
Pyrene	EPA 8270D/625	EPA 8270D
Total Petroleum Hydrocarbons (TPH)	FL-PRO	FL-PRO
<u>Volatile Organics</u>		
1,1,1,2-Tetrachloroethane	EPA 8260B/624	EPA 8260B
1,1,1-Trichloroethane	EPA 8260B/624	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B/624	EPA 8260B
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260B/624	EPA 8260B
1,1,2-Trichloroethane	EPA 8260B/624	EPA 8260B
1,1-Dichloroethane	EPA 8260B/624	EPA 8260B
1,1-Dichloroethene	EPA 8260B/624	EPA 8260B
1,1-Dichloropropene	EPA 8260B/624	EPA 8260B
1,2,3-Trichlorobenzene	EPA 504.1/8260B/624	EPA 8260B
1,2,3-Trichloropropane	EPA 8260B/624	EPA 8260B
1,2,4-Trichlorobenzene	EPA 8260B/624	EPA 8260B
1,2,4-Trimethylbenzene	EPA 8260B/624	EPA 8260B
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504 /504.1/8011/8260B	EPA 8260B
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504 /504.1/8011/8260B	EPA 8260B
1,2-Dichlorobenzene	EPA 8260B/624	EPA 8260B
1,2-Dichloroethane	EPA 8260B/624	EPA 8260B

Peter Abney

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
1,2-Dichloropropane	EPA 8260B/624	EPA 8260B
1,3,5-Trimethylbenzene	EPA 8260B/624	EPA 8260B
1,3-Dichlorobenzene	EPA 8260B/624	EPA 8260B
1,3-Dichloropropane	EPA 8260B/624	EPA 8260B
1,4-Dichlorobenzene	EPA 8260B/624	EPA 8260B
1,4-Dioxane (1,4-Diethylenoxide)	EPA 8260B/624	EPA 8260B
2,2-Dichloropropane	EPA 8260B/624	EPA 8260B
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260B/624	EPA 8260B
2-Chloroethyl vinyl ether	EPA 8260B/624	EPA 8260B
2-Chlorotoluene	EPA 8260B/624	EPA 8260B
2-Hexanone	EPA 8260B/624	EPA 8260B
4-Chlorotoluene	EPA 8260B/624	EPA 8260B
4-Methyl-2-pentanone (MIBK)	EPA 8260B/624	EPA 8260B
Acetone	EPA 8260B/624	EPA 8260B
Acetonitrile	EPA 8260B/624	EPA 8260B
Acrolein (Propenal)	EPA 8260B/624	EPA 8260B
Acrylonitrile	EPA 8260B/624	EPA 8260B
Allyl chloride (3-Chloropropene)	EPA 8260B/624	EPA 8260B
Benzene	EPA 8260B/624	EPA 8260B
Bromobenzene	EPA 8260B/624	EPA 8260B
Bromochloromethane	EPA 8260B/624	EPA 8260B
Bromodichloromethane	EPA 8260B/624	EPA 8260B
Bromoform	EPA 8260B/624	EPA 8260B
Carbon tetrachloride	EPA 8260B/624	EPA 8260B
Carbon disulfide	EPA 8260B/624	EPA 8260B
Chlorobenzene	EPA 8260B/624	EPA 8260B
Chloroethane	EPA 8260B/624	EPA 8260B
Chloroform	EPA 8260B/624	EPA 8260B
Chloroprene	EPA 8260B/624	EPA 8260B
cis-1,2-Dichloroethene	EPA 8260B/624	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B/624	EPA 8260B
Cyclohexane	EPA 8260B/624	EPA 8260B
Dibromochloromethane	EPA 8260B/624	EPA 8260B
Dibromomethane	EPA 8260B/624	EPA 8260B
Dichlorodifluoromethane	EPA 8260B/624	EPA 8260B
Ethyl methacrylate	EPA 8260B/624	EPA 8260B
Hexachlorobutadiene	EPA 8260B/624	EPA 8260B
Ethylbenzene	EPA 8260B/624	EPA 8260B
Iodomethane (Methyl iodide)	EPA 8260B/624	EPA 8260B
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260B/624	EPA 8260B
Isopropylbenzene	EPA 8260B/624	EPA 8260B
m+p-Xylenes	EPA 8260B/624	EPA 8260B
Methacrylonitrile	EPA 8260B/624	EPA 8260B
Methyl acetate	EPA 8260B/624	EPA 8260B
Methyl bromide (Bromomethane)	EPA 8260B/624	EPA 8260B
Methyl chloride (Chloromethane)	EPA 8260B/624	EPA 8260B
Methyl methacrylate	EPA 8260B/624	EPA 8260B
Methyl tert-butyl ether (MTBE)	EPA 8260B/624	EPA 8260B
Methylcyclohexane	EPA 8260B/624	EPA 8260B
Methylene chloride	EPA 8260B/624	EPA 8260B



Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
Naphthalene	EPA 8260B/624	EPA 8260B
n-Butylbenzene	EPA 8260B/624	EPA 8260B
n-Propylbenzene	EPA 8260B/624	EPA 8260B
o-Xylene	EPA 8260B/624	EPA 8260B
Pentachloroethane	EPA 8260B/624	EPA 8260B
p-Isopropyltoluene	EPA 8260B/624	EPA 8260B
Propionitrile (Ethyl cyanide)	EPA 8260B/624	EPA 8260B
sec-Butylbenzene	EPA 8260B/624	EPA 8260B
Styrene	EPA 8260B/624	EPA 8260B
tert-Butylbenzene	EPA 8260B/624	EPA 8260B
Tetrachloroethene (Perchloroethylene)	EPA 8260B/624	EPA 8260B
Toluene	EPA 8260B/624	EPA 8260B
trans-1,2-Dichloroethene	EPA 8260B/624	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B/624	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B/624	EPA 8260B
Trichloroethene (Trichloroethylene)	EPA 8260B/624	EPA 8260B
Trichlorofluoromethane	EPA 8260B/624	EPA 8260B
Vinyl acetate	EPA 8260B/624	EPA 8260B
Vinyl chloride	EPA 8260B/624	EPA 8260B
Xylene (total)	EPA 8260B/624	EPA 8260B
<u>Pesticides-Herbicides-PCBs</u>		
2,4,5-T	EPA 8151A /615	EPA 8151A
2,4-D	EPA 8151A /615	EPA 8151A
2,4-DB	EPA 8151A /615	EPA 8151A
3,5-Dichlorobenzoic acid	EPA 8151A /615	EPA 8151A
4,4'-DDD	EPA 8081B/608	EPA 8081B
4,4'-DDE	EPA 8081B/608	EPA 8081B
4,4'-DDT	EPA 8081B/608	EPA 8081B
4-Nitrophenol	EPA 8151A/615	EPA 8151A
Acifluorfen	EPA 8151A/615	EPA 8151A
Aldrin	EPA 8081B/608	EPA 8081B
alpha-BHC (alpha- Hexachlorocyclohexane)	EPA 8081B/608	EPA 8081B
alpha-Chlordane	EPA 8081B/608	EPA 8081B
Aroclor-1016(PCB-1016)	EPA 8082A/608	EPA 8082A
Aroclor-1221 (PCB-1221)	EPA 8082A/608	EPA 8082A
Aroclor-1232 (PCB-1232)	EPA 8082A/608	EPA 8082A
Aroclor-1242 (PCB-1242)	EPA 8082A/608	EPA 8082A
Aroclor-1248 (PCB-1248)	EPA 8082A/608	EPA 8082A
Aroclor-1254 (PCB-1254)	EPA 8082A/608	EPA 8082A
Aroclor-1260 (PCB-1260)	EPA 8082A/608	EPA 8082A
Azinphos-methyl (Guthion)	EPA 8141B/614	EPA 8141B
Bentazon	EPA 8151A/615	EPA 8151A
beta-BHC (beta- Hexachlorocyclohexane)	EPA 8081B/608	EPA 8081B
Bolstar (Sulprofos)	EPA 8141B/614	EPA 8141B
Chloramben	EPA 8151A/615	EPA 8151A
Chlordane (tech.)	EPA 8081B/608	EPA 8081B
Chlorpyrifos	EPA 8141B/614	EPA 8141B
Coumaphos	EPA 8141B/614	EPA 8141B

Peter Abney

Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
Dacthal (DCPA)	EPA 8151A/615	EPA 8151A
Dalapon	EPA 8151A/615	EPA 8151A
delta-BHC	EPA 8081B/608	EPA 8081B
Demeton-o	EPA 8141B/614	EPA 8141B
Demeton-s	EPA 8141B/614	EPA 8141B
Diazinon	EPA 8141B/614	EPA 8141B
Dicamba	EPA 8151A/615	EPA 8151A
Dichlorofenthion	EPA 8141B/614	EPA 8141B
Dichloroprop (Dichlorprop)	EPA 8151A/615	EPA 8151A
Dlchlorovos (DDVP, Dichtorvos)	EPA 8141B/614	EPA 8141B
Dieldrin	EPA 8081B/608	EPA 8081B
Dimethoate	EPA 8141B/614	EPA 8141B
Dinoseb (2-sec-buty1-4 ,6-dinilrophenol, DNB P)	EPA 8151A/615	EPA 8151A
Disulfoton	EPA 8141B/614	EPA 8141B
Endosulfan I	EPA 8081B/608	EPA 8081B
Endosulfan II	EPA 8081B/608	EPA 8081B
Endosulfan sulfate	EPA 8081B/608	EPA 8081B
Endrin	EPA 8081B/608	EPA 8081B
Endrin aldehyde	EPA 8081B/608	EPA 8081B
Endrin ketone	EPA 8081B/608	EPA 8081B
EPN	EPA 8141B/614	EPA 8141B
Ethion	EPA 8141B/614	EPA 8141B
Ethoprop	EPA 8141B/614	EPA 8141B
fensulfothion	EPA 8141B/614	EPA 8141B
fenthion	EPA 8141B/614	EPA 8141B
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081B/608	EPA 8081B
gamma-Chlordane	EPA 8081B/608	EPA 8081B
Heptachlor	EPA 8081B/608	EPA 8081B
Heptachlor epoxide	EPA 8081B/608	EPA 8081B
Isodrin	EPA 8081B/608	EPA 8081B
Malathion	EPA 8141B/614	EPA 8141B
MCPA	EPA 8151A/615	EPA 8151A
MCPP	EPA 8151A/615	EPA 8151A
Merphos	EPA 8141B/614	EPA 8141B
Methoxychlor	EPA 8081B/608	EPA 8081B
Methyl parathion (Parathion. methyl)	EPA 8141B/614	EPA 8141B
Mevinphos	EPA 8141B/614	EPA 8141B
Mirex	EPA 8081B/608	EPA 8081B
Monocrotophos	EPA 8141B/614	EPA 8141B
Naled	EPA 8141B/614	EPA 8141B
Parathion, ethyl	EPA 8141B/614	EPA 8141B
Pentachlorophenol	EPA 8151A/615	EPA 8151A
Phorate	EPA 8141B/614	EPA 8141B
Picloram	EPA 8151A/615	EPA 8151A
Ronnel	EPA 8141B/614	EPA 8141B
Silvex (2A.5-TP)	EPA 8151B/615	EPA 8151B
Stirofos	EPA 8141B/614	EPA 8141B
Sulfotepp	EPA 8141B/614	EPA 8141B
Tetraethyl pyrophosphate (TEPP)	EPA 8141B/614	EPA 8141B
Tokuthion (Prothiophos)	EPA 8141B/614	EPA 8141B



Analyte / Parameter	Non-Potable Water	Solid Hazardous Waste
Toxaphene (Chlorinated camphene)	EPA 8081B/608	EPA 8081B
Trichloronate	EPA 8141B/614	EPA 8141B

Preparation Methods

Fraction	Analytical Method	Preparation Method
Cyanide	EPA 9014 EPA 335.2 /SM 4500-CN E	EPA 9010C
TX	EPA 9056A	EPA 5050
Metal water prep	EPA 6020A/200.8	EPA 3005A
Metals soil prep	EPA 6020A	EPA 3050B
Metals TCLP prep	EPA 6020A/200.8	EPA 3010A
Extractable organics and Pesticides water prep	EPA 8270D/625/8081B/8082A/ 608/ 8141B/ 614	EPA 3510C
Extractable organics and Pesticides waste prep	EPA 8270D/625/8081B/8082A/ 608/ 8141B/ 614	EPA 3580A
Extractable organics and Pesticides soil prep	EPA 8270D/625/8081B/8082A/ 608/ 8141B/ 614	EPA 3550C
Organics water and mid-level soil prep	EPA 8260B/624	EPA 5030B
Organics low-level soil prep	EPA 8260B/624	EPA 5035
Soil/water leachate	Wets	ENCO WETS-88
SPLP	Wets, Organics, and Metals	EPA 1312
TCLP	Wets, Organics, and Metals	EPA 1311



The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

ENVIRONMENTAL CONSERVATION LABORATORIES - ORLANDO

Orlando, FL

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).



Presented this 29th day of March 2010.

A handwritten signature in black ink, appearing to read "Peter Meyer".

President & CEO
For the Accreditation Council
Certificate Number 3000.01
Valid to March 31, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.



State of Florida
Department of Health, Bureau of Laboratories
This is to certify that

E83182

ENVIRONMENTAL CONSERVATION LABORATORIES, INC. (ENCO) -
ORLANDO
10775 CENTRAL PORT DRIVE
ORLANDO, FL 32824-7009

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, 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.

EFFECTIVE August 22, 2010 THROUGH June 30, 2011



A handwritten signature in black ink that reads "Max Salfinger".

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04

NON-TRANSFERABLE E83182-26-08/22/2010
Supersedes all previously issued certificates

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 1 of 27

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Bromide	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2009
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	7/30/2009
Dissolved organic carbon (DOC)	SM 5310 B	Primary Inorganic Contaminants	NELAP	7/30/2009
Escherichia coli	COLISURE	Microbiology	NELAP	10/1/2008
Fluoride	EPA 300.0	Primary Inorganic Contaminants	NELAP	3/30/2010
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2009
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2009
Odor	SM 2150 B	Secondary Inorganic Contaminants	NELAP	7/30/2009
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2009
Orthophosphate as P	EPA 365.1	Primary Inorganic Contaminants	NELAP	7/30/2009
pH	SM 4500-H+-B	Secondary Inorganic Contaminants	NELAP	7/30/2009
Sulfate	EPA 300.0	Primary Inorganic Contaminants	NELAP	3/30/2010
Surfactants - MBAS	SM 5540 C	Secondary Inorganic Contaminants	NELAP	7/30/2009
Total coliforms	COLISURE	Microbiology	NELAP	10/1/2008
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2009
Total organic carbon	SM 5310 B	Primary Inorganic Contaminants	NELAP	7/30/2009
UV 254	SM 5910 B	Primary Inorganic Contaminants	NELAP	7/30/2009

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260	Volatile Organics	NELAP	11/17/2006
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/13/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 504.1	Volatile Organics	NELAP	4/27/2006
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504	Volatile Organics	NELAP	2/13/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Volatile Organics	NELAP	4/27/2006
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504	Volatile Organics	NELAP	2/13/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Volatile Organics	NELAP	4/27/2006
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2002
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	2/13/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 3 of 27

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	6/18/2009
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2002
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2002
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,5-T	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-D	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	2/13/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	2/13/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	2/13/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	2/13/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 625	Extractable Organics	NELAP	11/17/2006
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	2/13/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,5-Dichlorobenzoic acid	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	11/17/2006
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	9/17/2002

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Laboratory Scope of Accreditation

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	2/13/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	2/13/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	2/13/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
4-Nitrophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	2/13/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	2/13/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	2/13/2002
Acidity, as CaCO3	SM 2310 B (4A)	General Chemistry	NELAP	2/4/2008
Acifluorfen	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	2/13/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	2/13/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	2/13/2002
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	10/30/2002
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	2/4/2008
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aluminum	EPA 200.8	Metals	NELAP	4/14/2006
Aluminum	EPA 6020	Metals	NELAP	4/14/2006
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	2/13/2002
Amenable cyanide	EPA 9010/9014	General Chemistry	NELAP	7/1/2003
Ammonia as N	EPA 350.1	General Chemistry	NELAP	2/13/2002
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	2/13/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.8	Metals	NELAP	4/14/2006
Antimony	EPA 6020	Metals	NELAP	4/14/2006
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 200.8	Metals	NELAP	4/14/2006
Arsenic	EPA 6020	Metals	NELAP	4/14/2006
Atrazine	EPA 8270	Extractable Organics	NELAP	10/1/2008
Azinphos-methyl (Guthion)	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006

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Laboratory Scope of Accreditation

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Barium	EPA 200.8	Metals	NELAP	4/14/2006
Barium	EPA 6020	Metals	NELAP	4/14/2006
Bentazon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Benzaldehyde	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
Benzene	EPA 624	Volatile Organics	NELAP	2/13/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzidine	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.8	Metals	NELAP	4/14/2006
Beryllium	EPA 6020	Metals	NELAP	4/14/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	2/13/2002
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	2/4/2008
Biphenyl	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	2/13/2002
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	2/13/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	2/13/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003

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(407) 826-5314

E83182
Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	2/13/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	2/13/2002
Bromide	EPA 9056	General Chemistry	NELAP	7/1/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	2/13/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	EPA 624	Volatile Organics	NELAP	2/13/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 200.8	Metals	NELAP	4/14/2006
Cadmium	EPA 6020	Metals	NELAP	4/14/2006
Calcium	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Calcium	EPA 6020	Metals	NELAP	4/14/2006
Caprolactam	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	2/13/2002
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	2/13/2002
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	2/13/2002
Chloramben	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	2/13/2002
Chloride	EPA 9056	General Chemistry	NELAP	7/1/2003
Chlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	2/13/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	EPA 624	Volatile Organics	NELAP	2/13/2002

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E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorophylls	SM 10200 H	General Chemistry	NELAP	10/1/2008
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 200.8	Metals	NELAP	4/14/2006
Chromium	EPA 6020	Metals	NELAP	4/14/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	2/13/2002
Chrysene	EPA 625	Extractable Organics	NELAP	2/13/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	2/13/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.8	Metals	NELAP	4/14/2006
Cobalt	EPA 6020	Metals	NELAP	4/14/2006
Color	EPA 110.2	General Chemistry	NELAP	2/13/2002
Color	SM 2120 B	General Chemistry	NELAP	2/4/2008
Conductivity	EPA 120.1	General Chemistry	NELAP	2/13/2002
Copper	EPA 200.8	Metals	NELAP	4/14/2006
Copper	EPA 6020	Metals	NELAP	4/14/2006
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Cyanide	EPA 335.2	General Chemistry	NELAP	2/13/2002
Cyanide	SM 4500-CN E	General Chemistry	NELAP	2/4/2008
Cyclohexane	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Dacthal (DCPA)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dalapon	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Demeton-o	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Demeton-s	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Demeton-s	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diazinon	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006

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Issue Date: 8/22/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	2/13/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	2/13/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorofenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichloroprop (Dichlorprop)	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethoate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Disulfoton	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/1/2008
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002

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EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
EPN	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethion	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Ethion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	2/13/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9222 D	Microbiology	NELAP	7/30/2009
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ferric iron (calc.)	SM 3500-Fe D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	11/17/2006
Ferrous iron	SM 3500-Fe D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	11/17/2006
Fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	2/13/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	2/13/2002
Fluoride	EPA 9056	General Chemistry	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hardness	EPA 130.2	General Chemistry	NELAP	2/13/2002
Hardness	SM 2340 B	General Chemistry	NELAP	2/4/2008
Hardness	SM 2340 C	General Chemistry	NELAP	7/30/2009

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	2/13/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	2/13/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	2/13/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	2/13/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Iron	EPA 6020	Metals	NELAP	4/14/2006
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Isophorone	EPA 625	Extractable Organics	NELAP	2/13/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	2/13/2002
Lead	EPA 200.8	Metals	NELAP	4/14/2006
Lead	EPA 6020	Metals	NELAP	4/14/2006
m+p-Xylenes	EPA 8260	Volatile Organics	NELAP	10/1/2008
Magnesium	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Magnesium	EPA 6020	Metals	NELAP	4/27/2006
Malathion	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006

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Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Manganese	EPA 200.8	Metals	NELAP	4/14/2006
Manganese	EPA 6020	Metals	NELAP	4/14/2006
MCPA	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mercury	EPA 245.1	Metals	NELAP	8/28/2006
Mercury	EPA 7470	Metals	NELAP	4/27/2006
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 608.2	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl acetate	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	2/13/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	2/13/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylcyclohexane	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Methylene chloride	EPA 624	Volatile Organics	NELAP	2/13/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 200.8	Metals	NELAP	4/14/2006
Molybdenum	EPA 6020	Metals	NELAP	4/14/2006
Monocrotophos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Naled	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Naphthalene	EPA 625	Extractable Organics	NELAP	2/13/2002

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E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.8	Metals	NELAP	4/14/2006
Nickel	EPA 6020	Metals	NELAP	4/14/2006
Nitrate	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	2/13/2002
Nitrate as N	EPA 353.1	General Chemistry	NELAP	2/13/2002
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	2/13/2002
Nitrate-nitrite	EPA 353.1	General Chemistry	NELAP	2/13/2002
Nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrite	SM 4500-NO2 B	General Chemistry	NELAP	2/4/2008
Nitrite as N	EPA 300.0	General Chemistry	NELAP	2/13/2002
Nitrite as N	EPA 354.1	General Chemistry	NELAP	2/13/2002
Nitrobenzene	EPA 625	Extractable Organics	NELAP	2/13/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	2/13/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	2/13/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	10/30/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Organic nitrogen	EPA 351.2 - EPA 350.1	General Chemistry	NELAP	2/13/2002
Orthophosphate as P	EPA 365.1	General Chemistry	NELAP	10/30/2002
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	2/13/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
o-Xylene	EPA 8260	Volatile Organics	NELAP	11/17/2006

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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E83182

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10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Parathion, ethyl	EPA 614	Pesticides-Herbicides-PCB's	NELAP	11/17/2006
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	3/12/2010
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	10/1/2008
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2002
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
pH	EPA 150.1	General Chemistry	NELAP	2/13/2002
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	2/4/2008
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	2/13/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	2/13/2002
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	2/13/2002
Picloram	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Potassium	EPA 6020	Metals	NELAP	4/14/2006
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pyrene	EPA 625	Extractable Organics	NELAP	2/13/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	2/13/2002
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	2/4/2008
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	2/13/2002
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	2/4/2008
Residue-total	EPA 160.3	General Chemistry	NELAP	2/13/2002
Residue-total	SM 2540 B	General Chemistry	NELAP	2/4/2008
Residue-total	SM 2540 G	General Chemistry	NELAP	11/17/2006

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Issue Date: 8/22/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Residue-volatile	EPA 160.4	General Chemistry	NELAP	2/13/2002
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.8	Metals	NELAP	4/14/2006
Selenium	EPA 6020	Metals	NELAP	4/14/2006
Silver	EPA 200.8	Metals	NELAP	4/14/2006
Silver	EPA 6020	Metals	NELAP	4/14/2006
Silvex (2,4,5-TP)	EPA 615	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Sodium	EPA 6020	Metals	NELAP	4/27/2006
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	2/13/2002
Sulfate	EPA 9056	General Chemistry	NELAP	7/1/2003
Sulfide	EPA 376.1	General Chemistry	NELAP	2/13/2002
Sulfide	SM 4500-S E (18th Ed.)/TITR	General Chemistry	NELAP	2/4/2008
Sulfotep	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Surfactants - MBAS	EPA 425.1	General Chemistry	NELAP	2/13/2002
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	2/4/2008
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	2/13/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetraethyl pyrophosphate (TEPP)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Thallium	EPA 200.8	Metals	NELAP	4/14/2006
Thallium	EPA 6020	Metals	NELAP	4/14/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Tin	ENCO SOP MET/15.0/ICP-MS	Metals	NELAP	11/17/2006
Tin	EPA 6020	Metals	NELAP	4/14/2006
Titanium	EPA 6020	Metals	NELAP	10/1/2008
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Toluene	EPA 624	Volatile Organics	NELAP	2/13/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Total coliforms	SM 9222 B	Microbiology	NELAP	7/30/2009
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	7/1/2003
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Total nitrate-nitrite	SM 4500-NO3 H	General Chemistry	NELAP	2/4/2008
Total nitrogen	TKN + Total nitrate-nitrite	General Chemistry	NELAP	7/30/2009
Total organic carbon	EPA 415.1	General Chemistry	NELAP	4/27/2006
Total organic carbon	EPA 9060	General Chemistry	NELAP	11/17/2006
Total organic carbon	SM 5310 B	General Chemistry	NELAP	2/4/2008
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/1/2003
Total phenolics	EPA 420.1	General Chemistry	NELAP	2/13/2002
Total, fixed, and volatile residue	SM 2540 G	General Chemistry	NELAP	10/30/2002
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/13/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	2/13/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	2/13/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	2/13/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Turbidity	EPA 180.1	General Chemistry	NELAP	2/13/2002
Un-ionized Ammonia	DEP SOP 10/03/83	General Chemistry	NELAP	2/13/2002
Vanadium	EPA 200.8	Metals	NELAP	4/14/2006
Vanadium	EPA 6020	Metals	NELAP	4/14/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	2/13/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 624	Volatile Organics	NELAP	10/30/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Zinc	EPA 200.8	Metals	NELAP	4/14/2006
Zinc	EPA 6020	Metals	NELAP	4/14/2006

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State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1,2-Trichloro-1,2,2-trifluoroethane	EPA 8260	Volatile Organics	NELAP	11/17/2006
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/30/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	10/30/2002
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	6/18/2009
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	6/18/2009
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	10/30/2002
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	2/13/2002
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2002
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

**Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	2/13/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	2/13/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/13/2002
2-Hexanone	EPA 8260	Volatile Organics	NELAP	2/13/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	10/30/2002
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	2/13/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/30/2002
3,5-Dichlorobenzoic acid	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	11/17/2006
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/30/2002
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	2/13/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	2/13/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	2/13/2002

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182
Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	2/13/2002
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/13/2002
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/30/2002
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	10/30/2002
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	9/9/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2002
4-Nitrophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2002
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/30/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Acetone	EPA 8260	Volatile Organics	NELAP	2/13/2002
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/30/2002
Acetophenone	EPA 8270	Extractable Organics	NELAP	2/13/2002
Acifluorfen	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	2/13/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	11/17/2006
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	11/17/2006
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	7/30/2009
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	10/30/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Aluminum	EPA 6020	Metals	NELAP	4/14/2006
Ammonia as N	EPA 350.1	General Chemistry	NELAP	11/17/2006
Aniline	EPA 8270	Extractable Organics	NELAP	2/13/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Antimony	EPA 6020	Metals	NELAP	4/14/2006
Aramite	EPA 8270	Extractable Organics	NELAP	10/30/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002

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Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Arsenic	EPA 6020	Metals	NELAP	4/14/2006
Atrazine	EPA 8270	Extractable Organics	NELAP	10/1/2008
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Barium	EPA 6020	Metals	NELAP	4/14/2006
Bentazon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Benzaldehyde	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
Benzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Benzoic acid	EPA 8270	Extractable Organics	NELAP	10/30/2002
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	2/13/2002
Beryllium	EPA 6020	Metals	NELAP	4/14/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Biphenyl	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	2/13/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	2/13/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	2/13/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	2/13/2002
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Bromide	EPA 9056	General Chemistry	NELAP	2/13/2002
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/30/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	2/13/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Cadmium	EPA 6020	Metals	NELAP	4/14/2006
Calcium	EPA 6020	Metals	NELAP	4/27/2006

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State Laboratory ID: E83182 EPA Lab Code: FL00288 (407) 826-5314

**E83182
Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Caprolactam	ENCO SOP SVGCMS-03/GC-MS	Extractable Organics	NELAP	7/30/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	10/30/2002
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	2/13/2002
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/13/2002
Chloramben	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Chloride	EPA 9056	General Chemistry	NELAP	2/13/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
Chloroform	EPA 8260	Volatile Organics	NELAP	2/13/2002
Chloroprene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Chromium	EPA 6020	Metals	NELAP	4/14/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	10/7/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	2/13/2002
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Cobalt	EPA 6020	Metals	NELAP	4/14/2006
Copper	EPA 6020	Metals	NELAP	4/14/2006
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Cyclohexane	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Dacthal (DCPA)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Demeton-s	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	2/13/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/30/2002
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/13/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 23 of 27

Attachment to Certificate #: E83182-26, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorofenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Dimethoate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Diphenylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	3/11/2004
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/5/2010
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
EPN	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Ethion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/30/2002
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/13/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Fluoride	EPA 9056	General Chemistry	NELAP	2/13/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002

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Issue Date: 8/22/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E83182

EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	2/13/2002
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/30/2002
Iron	EPA 6020	Metals	NELAP	4/14/2006
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/30/2002
Isodrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	2/13/2002
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Isosafrole	EPA 8270	Extractable Organics	NELAP	2/13/2002
Keponc	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	11/17/2006
Lead	EPA 6020	Metals	NELAP	4/14/2006
m+p-Xylenes	EPA 8260	Volatile Organics	NELAP	11/17/2006
Magnesium	EPA 6020	Metals	NELAP	4/14/2006
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Manganese	EPA 6020	Metals	NELAP	4/14/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Mercury	EPA 7471	Metals	NELAP	8/28/2006
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/30/2002
Methapyrilene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Methyl acetate	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/30/2002
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/13/2002

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EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando

10775 Central Port Drive

Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Methylcyclohexane	ENCO SOP VGCMS/5.0/GC-MS	Volatile Organics	NELAP	11/17/2006
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/13/2002
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Molybdenum	EPA 6020	Metals	NELAP	4/14/2006
Monocrotophos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Naled	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Naphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Nickel	EPA 6020	Metals	NELAP	4/14/2006
Nitrate	EPA 9056	General Chemistry	NELAP	2/13/2002
Nitrate as N	EPA 353.1	General Chemistry	NELAP	11/17/2006
Nitrite	EPA 9056	General Chemistry	NELAP	2/13/2002
Nitrite	SM 4500-NO2 B	General Chemistry	NELAP	7/30/2009
Nitrite as N	EPA 354.1	General Chemistry	NELAP	11/17/2006
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/30/2002
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/30/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	10/30/2002
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/30/2002
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/30/2002
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2002
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	11/17/2006
o-Toluidine	EPA 8270	Extractable Organics	NELAP	2/13/2002
o-Xylene	EPA 8260	Volatile Organics	NELAP	11/17/2006
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002

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Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E83182 EPA Lab Code: FL00288 (407) 826-5314

E83182
Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/5/2010
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	10/1/2008
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	10/30/2002
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
pH	EPA 9040	General Chemistry	NELAP	2/13/2002
pH	EPA 9045	General Chemistry	NELAP	2/13/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	2/13/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Phenol	EPA 8270	Extractable Organics	NELAP	2/13/2002
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/5/2010
Phosphorus, total	EPA 365.1	General Chemistry	NELAP	11/17/2006
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	11/17/2006
Picloram	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Potassium	EPA 6020	Metals	NELAP	4/14/2006
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/30/2002
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/30/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2002
Pyridine	EPA 8270	Extractable Organics	NELAP	2/13/2002
Residue-total	EPA 160.3	General Chemistry	NELAP	11/17/2006
Residue-total	SM 2540 G	General Chemistry	NELAP	11/17/2006
Residue-volatile	EPA 160.4	General Chemistry	NELAP	11/17/2006
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Safrole	EPA 8270	Extractable Organics	NELAP	2/13/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Selenium	EPA 6020	Metals	NELAP	4/14/2006
Silver	EPA 6020	Metals	NELAP	4/14/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Sodium	EPA 6020	Metals	NELAP	4/27/2006
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Strontium	EPA 6020	Metals	NELAP	4/27/2006
Styrene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Sulfate	EPA 9056	General Chemistry	NELAP	2/13/2002

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Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Zinc	EPA 6020	Metals	NELAP	4/27/2006

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EPA Lab Code: FL00288

(407) 826-5314

E83182

Environmental Conservation Laboratories, Inc. (ENCO) - Orlando
10775 Central Port Drive
Orlando, FL 32824-7009

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sulfotep	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	2/13/2002
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Tetraethyl pyrophosphate (TEPP)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Thallium	EPA 6020	Metals	NELAP	4/14/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/30/2002
Tin	EPA 6020	Metals	NELAP	4/14/2006
Titanium	EPA 6020	Metals	NELAP	4/14/2006
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Toluene	EPA 8260	Volatile Organics	NELAP	2/13/2002
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	2/13/2002
Total nitrate-nitrite	EPA 353.1	General Chemistry	NELAP	11/17/2006
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	2/13/2002
Total nitrate-nitrite	SM 4500-NO3 H	General Chemistry	NELAP	7/30/2009
Total nitrogen	TKN + Total nitrate-nitrite	General Chemistry	NELAP	11/17/2006
Total organic carbon	WALKLEY-BLACK	General Chemistry	NELAP	5/5/2010
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	10/30/2002
Total phenolics	EPA 420.1	General Chemistry	NELAP	11/17/2006
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	2/13/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/13/2002
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/30/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/13/2002
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	2/13/2002
Vanadium	EPA 6020	Metals	NELAP	4/14/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	2/13/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/13/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/13/2002
Zinc	EPA 6020	Metals	NELAP	4/27/2006



ENVIRONMENTAL CONSERVATION LABORATORIES, INC

SOP No.	VGCMS-10		
Revision No.	1		
Effective Date	Nov 24, 2010		
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**Environmental Conservation Laboratories Inc.
STANDARD OPERATING PROCEDURE**

ANALYSIS OF 1,4 -DIOXANE BY GC-MS (SIM MODE).

Signed for:

Corporate

Operations

QA

NA

NA

Cary

Operations

QA

NA

NA

Jacksonville

Operations

QA

Orlando

Operations

QA

Each approved signatory will electronically sign with a facsimile of signature and the date of signing

Proprietary Information Statement:

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ENVIRONMENTAL CONSERVATION LABORATORIES, INC

SOP No.	VGCMS-10		
Revision No.	1		
Effective Date	Nov 24, 2010		
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1 Method

SW-846 8260B, SW-846 8260C, SW-846 5030B, SW-846 5035

2 MATRIX

Water, solid and waste

3 DETECTION LIMITS

Method detection limits (MDLs) and method reporting limits (MRLs) are maintained in LIMS. The method reporting limit (MRL) for these tests is Table 1, but is subject to change without updating this SOP.

4 SCOPE

This is a purge and trap gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of 1,4-Dioxane by SIM mode.

5 SUMMARY

The volatile compounds are introduced into the gas chromatograph by purge-and-trap techniques.

The analytes are introduced directly into a narrow-bore capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC). Analytes eluted from the capillary column are introduced directly into the mass spectrometer.

Identification of target analytes is accomplished by comparing their retention time and mass ion trace with those of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum five-point calibration curve.

6 DEFINITIONS

- 6.1 SIM Mode – Mass spectrometer mode where during each scan, only the ions specified are monitored.
- 6.2 Batch - a group of samples which are prepared together using the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch must be of similar composition (matrix).

Policy QA-001 specifies that the maximum batch size is 20 samples unless otherwise specified in the source method. The maximum time between start of processing of the first and last sample in the batch is 24 hours.
- 6.3 Sequence – a group of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. A sequence can include prepared samples originating from more than one batch and has no limit as to the



SOP No.	VGCMS-10		
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number of samples, provided frequency of calibration requirements in the cited method are followed.

- 6.4 Method Blank – (MB) contaminant free water, sand, teflon chips, glass beads or other appropriate matrix, taken through the entire analytical process to determine if there is any contamination associated with the analytical procedures.
- 6.5 Laboratory Control Sample (LCS) - contaminant free water, sand, Teflon chips glass beads or other appropriate matrix, spiked with known amounts of a subset or totality of the target compound, and taken through the entire analytical process to determine if there is any bias introduced by the sample preparation and analysis procedures.
- 6.6 Matrix spike (MS) - a sample prepared by spiking a known amount of the target analyte(s) to a sample for which the native concentration of target analyte concentration is known. Matrix spikes can be used to evaluate the effect of the sample matrix on the sample's analytical results. Matrix spike accuracy limits are based on Laboratory Control Sample limits unless otherwise specified by the source method.
- 6.7 Matrix spike duplicate (MSD) - a duplicate matrix spike is prepared to obtain a measure of the precision of the recovery for each analyte by comparing its results with those of the Matrix Spike.
- 6.8 Sample Duplicate – for procedures where matrix spiking is not viable, a second aliquot of a selected sample is analyzed and its result compared to the results of the first sample to assess precision of the analysis.
- 6.9 DI Water – Deionized water, whose quality is monitored according to SOP QAQC-15, "Deionized water monitoring."
- 6.10 Surrogate – compound which is not target analytes, which are added to samples to assess analytical performance of method designated to measure organic compounds. These compounds are spiked into all blanks, samples and spiked samples during sample preparation. Percent recoveries are calculated for each surrogate.

7 INTERFERENCES

- 7.1 Major contaminant sources are volatile materials (including organic solvents) in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.
- 7.2 Analyses of calibration and reagent blanks provide information about the presence of contaminants. Subtracting or deleting blank values from sample results is not permitted.
- 7.3 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After the analysis of a sample



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containing high concentrations of volatile organic compounds, one or more instrument blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

- 7.4 Samples can have high levels of volatile organic compounds (such as cis-1,2-dichloroethene) that can interfere with the internal standard 1,4-difluorobenzene. In these cases, the sample will need to be diluted in order to have acceptable internal standard recoveries.
- 7.5 It has been shown that 1,4-Dioxane is a poor purger at room temperature. In order get good recoveries out of the water, It needs to be heated while purging. The purge temperature used is 80°C. Clean up rinses also need to be heated.

8 SAFETY

- 8.1 Lab coats, safety glasses, and latex, nitrile or vinyl gloves are required to perform this analysis. Environmental samples are to be considered biological and chemical unknowns.
- 8.2 Hypodermic needles should be handled with care to prevent injury. Syringes with needles should be stored in a way that minimizes the hazard.

9 EQUIPMENT AND SUPPLIES

- 9.1 Analytical equipment
 - 9.1.1 Tekmar 3100 purge and trap concentrator, or equivalent
 - 9.1.2 EST Archon autosampler, or equivalent
 - 9.1.3 Gas chromatograph (HP 5890, Agilent 6890, or equivalent) interfaced with a mass selective detector (HP 5972, Agilent 5973, or equivalent)
 - 9.1.4 Data system to control instrument analysis parameters and process analytical results (HP/Agilent Chem Station, or equivalent)
 - 9.1.5 Analytical column: Restek RTX-VMS 60-m X 0.25-mm ID with a 1.4-um film thickness, or equivalent.
 - 9.1.6 Analytical trap: Supelco Vocab 3000 purge trap K, or equivalent.
- 9.2 Instrument settings –Times and temperatures are optimized for performance starting with the SIM Mode ion dwell times listed in **Table 6** and the SIM Mode temperature programming listed in **Table 7**.
- 9.3 Preservation and support tests:
 - 9.3.1 pH test strips: EMD pH 0-14 test strips, cat #9590, or equivalent
 - 9.3.2 Free Chlorine test strips: Aquachek Total chlorine/Free chlorine test strips, cat #27450-50, or equivalent



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- 9.4 Syringes, gas tight and luer tip
- 9.5 Volumetric glassware, Class A
- 9.6 VOA vials, 40-mL (nominal) vials, Greenwood, APC1601E, or equivalent
- 9.7 Washed Sand: VWR-Cat # BDH0274-2.5kg, or equivalent
- 9.8 Magnetic Stir Bars: Daniels Scientific-P/N-N000822, or equivalent
- 9.9 All maintenance is recorded in the instrument maintenance log in LIMS. Basic maintenance includes, cleaning the source, replacing the trap and mcs loop and will be performed as needed.
- 9.10 For troubleshooting the instrument, refer to the instrument manual.

10 REAGENTS AND STANDARDS

All reagents and standards are labeled with their unique ID, the name of the material, the date prepared and the expiration date. Preparation is documented in the Element laboratory information management system. Reagents are stored according to manufacturer's recommendations.

10.1 Reagents

- 10.1.1 Methanol, purge and trap quality
- 10.1.2 Reagent-grade water as required in SOP QAQC-15

10.2 Standards

The holding time for purchased stock solutions is the manufacturer's expiration date. The standard is stored between -1°C and -20°C.

The holding time for laboratory-prepared standards is 60 days. The standard is stored between -1°C and -20°C.

- 10.2.1 Tuning stock standard, 4-Bromofluorobenzene, 250 ug/mL in Methanol.
- 10.2.2 Stock Solutions: Stock solutions are generally purchased as certified solutions from commercial sources with certificates of analysis. These standards are stored in Teflon-sealed screw-cap bottles.
- 10.2.3 Working standards: A working solution containing the compounds of interest is prepared from the stock solution(s). The working solution for 1,4-Dioxane is prepared at 50ug/mL using Methanol as the solvent.
- 10.2.4 Second-source standards are similar to calibration standards, but are from a completely different source. Second-source standards are used for spiking (LCS, MS/MSD). The second-source standard is prepared at 50ug/mL using Methanol as the solvent.
- 10.2.5 Internal standards mix: 1,4-Dioxane-d8 purchased at 2,000ug/mL with an intermediate at 50ug/mL in Methanol or equivalent and 1,4-Difluorobenzene, purchased at 10,000ug/mL with an intermediate at 5 ug/mL in Methanol, or equivalent.
- 10.2.6 Surrogate standards mix, purchased at 10,000ug/mL, Toluene-d8, , intermediate at 5 ug/mL in Methanol, or equivalent.



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11 SAMPLE COLLECTION, PRESERVATION AND STORAGE

11.1 Water samples:

- 11.1.1 Two or more nominal 40-mL glass screw cap VOA vials with Teflon-faced silicone septa are filled for each water sample. Additional vials are required for samples designated for MS/MSD analysis.
- 11.1.2 Water samples are preserved with 1:1 HCl. The acid delays microbial degradation of any target analytes, particularly aromatics that may be present in the samples.
- 11.1.3 All water samples are stored between 2°C and 6°C.
- 11.1.4 The holding time is 14 days from sampling to analysis for water samples that have a pH ≤ 2. If the pH of the sample exceeds this criterion, the holding time changes to 7 days.
- 11.1.5 Preservation must be checked and documented for each sample after it has been analyzed. Aqueous samples preserved with Sodium Thiosulfate must also be tested for the presence of free chlorine and the result documented. Should the presence of free chlorine be established, or pH found to exceed requirements, project management is notified via a Non-Conformance memo (NCM).

11.2 Soil samples:

- 11.2.1 Soil samples are collected mostly in 2 oz. or 4 oz. jars, but can be collected in pre-weighed vials. Pre-weighed soil samples require a minimum of three vials for each soil being analyzed. Two vials are supplemented with deionized water and magnetic stir bars, and the third sample is supplemented with methanol.
- 11.2.2 Soil samples prepared in water are frozen within 48 hours of sample collection. Soil samples prepared in methanol are stored between -1°C and -20°C.
- 11.2.3 The holding time is 14 days from sampling to the completion of analysis for soils.

11.3 Waste samples:

- 11.3.1 Waste samples can be collected in a variety of containers, including containers provided for other analyses.

12 QUALITY CONTROL

Method criteria for frequency of QC samples are listed in **Table 4**. Additional or replacement requirements as indicated by project-specific documentation will be followed.

- 12.1 DoD Quality Systems Manual supersedes all requirements. For additional information on DoD based projects reference SOP PI-001.
- 12.2 Each preparation batch must include a method blank. For this test, the method blank consists of a clean matrix (deionized water, sand) that has been supplemented with surrogates and purged using the same reagents and equipment as regular samples.



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- 12.3 Each preparation batch must include a laboratory control sample (LCS). For this test, the LCS (blank spike) consists of a clean matrix (deionized water, sand) that has been supplemented with a second source spike mix, surrogates and purged using the same reagents and equipment as regular samples.
- 12.4 Each preparation batch must include at least one matrix spike and matrix spike duplicate (MS/MSD) which is spiked with known amounts of the target analytes and prepared in the same way as the native sample. If there is insufficient sample to prepare an MS/MSD, sample preparation must prepare a duplicate LCS (LCSd).
- 12.5 Documentation requirements
- Analysis results are documented in standard quantitation reports provided by the data system. All results are electronically transferred into the LIMS. Additional documentation that is required to document manual integrations is described in policy QA-011, "Manual integration".
- Sample preparation is documented in the LIMS benchsheet (**Error! Reference source not found.1**). No additional documentation is necessary; however, a logbook still used in Orlando (**Error! Reference source not found.5**).

13 CALIBRATION AND STANDARDIZATION

- 13.1 Calibration procedures must follow the requirements of policy QA-008, "Calibration requirements".
- 13.2 DoD Quality Systems Manual supersedes all requirements. For additional information on DoD based projects reference SOP PI-001.
- 13.3 The procedure for analysis of calibration standards is identical to that described in the Procedure section of this SOP, therefore this sections considers only evaluation of the calibration procedures.
- 13.4 The analytical sequence specified in Table 3 is followed for all sequences.. Calibration and QC requirements are summarized in **Table 4**.
- 13.5 Project-specific requirements must be followed in all cases.
- 13.6 Mass spectrometer tune verification - The mass spectrometer tune is verified using BFB at the beginning of every tune period.
- 13.6.1 Analyze 50 ng on-column of 4-Bromofluorobenzene (BFB).
- 13.6.2 Obtain a mass spectrum of the BFB peak consisting of the average of the apex scan and the scans immediately before and after the apex. Subtract a scan no more than 20 scans prior to the elution of BFB. This averaged and background subtracted mass spectrum must meet the criteria listed in **Table 5**.
- 13.6.3 Not meeting this requirement requires corrective action as described in Section 17.
- 13.6.4 Once the tuning requirements are met, analyses can proceed uninterrupted for a maximum of the time period indicated in **Table 4**. The basis for evaluating tune clock compliance is analysis start time.
- 13.7 Initial calibration (ICAL) – An initial calibration curve is analyzed and evaluated before any samples can be analyzed.



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- 13.7.1 Analyze the initial calibration curve standards (Table 2).
- 13.7.2 Calculate the relative response factor (RRF, Equation 1) for each analyte in each calibration standard, then the average RRF (Equation 2) and standard deviation (Equation 3) of the RRF for each analyte across the calibration range. Finally, calculate the %RSD for each analyte (Equation 4).
- 13.7.3 Average calibration curves must have an RSD that does not exceed the criteria in Table 4.
- 13.7.4 Curves not meeting the average calibration curve criterion may be fitted to a linear regression curve. The coefficient of determination must be at least 0.990, and each individual calibration point must be recalculated using the new curve (**Error! Reference source not found.2**). The calculated concentration of each individual calibration point may not exceed the requirements expressed in policy QA-008.
- 13.8 Second-source calibration verification (SCV) - A second-source calibration verification standard is analyzed immediately after the initial calibration curve.
- 13.8.1 A standard is prepared from a source independent of the standards used to generate the curve, at a concentration equivalent to the CCV, unless otherwise specified in the source method.
- 13.8.2 Calculate %Recovery of each analyte in the standard, which must be within the criteria specified in Table 4.
- 13.8.3 Internal standard requirements specified in Table 4 must be met.
- 13.8.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or the second-source verification. Corrective action must be taken (including reanalysis of the second source standard, or analysis of a different second source standard). Any decision to proceed with analysis of samples when the SCV standard is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
- 13.9 Continuing calibration (CCV)
- 13.9.1 A calibration verification standard is analyzed before any samples in sequences that don't include initial calibrations. The concentration of the CCV standard is listed in Table 2.
- 13.9.2 Calculate the %Recovery of each analyte in the standard, which must be within the criteria specified in Table 4.
- 13.9.3 Internal standard requirements specified in Table 4 must be met.
- 14 PROCEDURE**
- 14.1 Prior to sample analysis, perform preventive maintenance as required in the LIMS Instrument maintenance log.
- 14.2 Allow samples and calibration standards to reach room temperature.



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- 14.3 Ensure that the autosampler has sufficient volume of the internal standard and surrogate spike solutions.
- 14.4 Create a sequence in Element. The name of the sequence will be created automatically when the sequence is saved. Ensure that the correct instrument and calibration are selected, as well as the STD ID and ISTD ID. Follow the guidance in Table 3.
- 14.5 Water samples

The default analysis volume for water samples is 15 mL. Purging can be performed by:

 - 14.5.1 An aliquot of sample equivalent to the default analysis volume is manually transferred to a VOA vial and sealed immediately. The sample is placed in the autosampler and is purged directly from this vial. Samples are purged at 80°C.
- 14.6 Low-level soil samples
 - 14.6.1 Low-level soils can be received in a 2 oz. or 4 oz. jar or in pre-weighed vial.
 - 14.6.1.1 For low-level soil samples received in 2 oz. or 4 oz. jars, approximately 5 grams of soil is weighed into a VOA vial and 10 ml of reagent water is added. The vials are then sealed immediately.
 - 14.6.1.2 Low-level soil samples are received in pre-weighed vials containing a magnetic stir bar and measured volume of reagent water. The sample is stored frozen, and must be allowed to thaw before commencing the procedure. 5 ml of reagent water must be added before purging can begin.
 - 14.6.2 Measure and record the final weight of the sample before water is added.
 - 14.6.3 The samples are purged at 80°C on a magnetic stirrer that is an integral part of the autosampler.
- 14.7 Mid-to-high level soils
 - 14.7.1 If a mid-to-high level is needed, it is better to analyze by scan mode. Check with project management before analysis.
 - 14.7.2 Mid-to-high level soil samples are received in 2 oz. or 4 oz. jars or as pre-weighed vials containing measured volume of methanol (10 mL).
 - 14.7.2.1 If a jar is received, approximately 5 grams of soil is weighed into a VOA vial and 10 mL of Methanol is added.
 - 14.7.3 Measure and record the final weight of each sample.
 - 14.7.4 Shake the methanol-soil sample dispersion vigorously.
 - 14.7.5 Transfer 1mL of the methanol dispersion into 50 mL of reagent water, proportioned as necessary, and then measure 15mL into a VOA vial. The sample is thereafter processed in the same manner as a regular water sample.
- 14.8 Start the analysis sequence.

Upon sequence initiation, the autosampler automatically adds the internal standard and surrogate spike solutions, and commences the purge cycle, alternately hand spiking is allowed.



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14.9 Once the samples have run process the data and upload into element.

15 DATA ANALYSIS AND CALCULATIONS

All calculations are performed by the instrument data system, custom spreadsheets or the LIMS. Raw results are uploaded directly into the LIMS, which calculates final sample results applying dilution factors, actual sample preparation weights and volumes, and soil moisture content. Recoveries are also calculated using LIMS functionality.

Many instrument data systems allow for the entry of MDLs, spike control limits and other variables. At ENCO, the only variables that the analyst can enter into the data system are those which are not updatable, such as CCV and initial calibration curve control limits.

15.1 Data review

15.1.1 Identified compounds are reviewed for proper integration. Manual integrations are performed only if necessary and are documented by the analyst with a copy of before and after chromatograms which clearly show the change made. Manual integrations are reviewed by a peer as required by policy QA-011 ("Manual integration").

15.1.2 Target compounds identified by the data system are evaluated using the criteria listed in Section 15.3.

15.2 Dilutions

15.2.1 Only water and mid-to-high level soil samples can be analyzed at dilution in this procedure.

15.2.2 If the response for any compound exceeds the working range of the analytical system, a dilution of the sample is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run.

15.2.3 If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

15.2.4 The most concentrated dilution with no target compounds above the calibration range will be reported. If multiple dilutions of a sample are analyzed, the analyst will select the best results from the various dilutions, using as a guideline the selection of the least dilute yet calibrated result.

15.2.5 If the sample is diluted due to non-target compounds present in the sample, an NCM must be written describing the problem.

15.2.6 To dilute a sample,

15.2.6.1 Water samples: Dilute an aliquot of the sample with reagent grade water to a final volume equivalent to the default sample analysis volume.

15.2.6.2 Mid-to-high level soils: Add a smaller aliquot of the methanol dispersion to the default sample analysis volume of reagent grade water.

15.2.6.3 The dilution factor is calculated using Equation 13.



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15.2.7 If the dilution for a sample requires that less than 10% of the syringe capacity be used for the sample extract, multiple dilutions must be used. The final dilution factor is the product of the multiple dilution factors. Thus, if the sample extract is diluted by a factor of 100, and the dilution is diluted by a factor of 10, the total dilution is 1,000 (100 x 10).

15.3 Target analyte identification confirmation

15.3.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

15.3.2 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard's component. For reference, the standard must be run within the same twelve hours as the sample.

15.3.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

15.3.4 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

15.3.5 The relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

15.3.6 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation. Compounds analyzed using selected ion monitoring (SIM) technology require that at least two ions be monitored, and that they have the same retention time to confirm the presence of the target compound.

15.4 Equations

Equation 1. Response factor (RRF) calculation.

$$RRF = \left(\frac{\text{Response of target analyte}}{\text{Concentration of target analyte}} \right) \times \left(\frac{\text{Concentration of internal standard}}{\text{Response of internal standard}} \right)$$

Equation 2. Average response factor.



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$$\overline{RF} = \frac{\sum_{n=1}^n RF_n}{n}$$

Equation 3. Standard deviation (SD).

$$SD = \sqrt{\frac{\sum_{n=1}^n (RF_n - \overline{RF})^2}{n-1}}$$

Equation 4. % relative standard deviation (%RSD).

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

Equation 5. % D (% Difference or % Drift).

$$\% D = \frac{C_{\text{expected}} - C_{\text{found}}}{C_{\text{expected}}} \times 100$$

Equation 6. Extract concentration by linear regression curve.

$$\text{Extract concentration } \left(\frac{\mu\text{g}}{\text{mL}} \right) = \frac{\text{Response} - \text{Intercept}}{\text{Slope}}$$

Equation 7. Extract concentration using average response factor.

$$\text{Raw Amount} = \left(\frac{\text{Response}}{\overline{RRF}} \right) \times \left(\frac{\text{Concentration of internal standard}}{\text{Response of internal standard}} \right)$$

Equation 8. Water Sample Concentration

$$\text{Sample Conc. } \left(\frac{\mu\text{g}}{\text{L}} \right) = \frac{\text{Extract concentration } \left(\frac{\mu\text{g}}{\text{mL}} \right) \times \text{Total Extract Vol (mL)} \times \text{Dilution Factor}}{\text{Sample Volume (L)}}$$

Equation 9. Low-level Solid Sample Concentration.

$$\text{Sample Conc. } \left(\frac{\mu\text{g}}{\text{Kg}} \right) = \frac{\text{Extract concentration } \left(\frac{\mu\text{g}}{\text{mL}} \right) \times \text{Total Extract Vol (mL)}}{\text{Sample Weight (Kg)} \times \left(\frac{\% \text{ solids}}{100} \right)}$$

Equation 10. LCS and surrogate recovery.

$$\% \text{Recovery} = \left(\frac{\text{Amount found}}{\text{Amount spiked}} \right) \times 100$$

Equation 11. MS/MSD recovery.

$$\text{Matrix Spike \% Recovery} = \left(\frac{\text{Amount found} - \text{Native amount}}{\text{Amount spiked}} \right) \times 100$$

Equation 12. Relative percent difference.



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$$RPD = \left(\frac{|\text{Dup1 Result} - \text{Dup2 Result}|}{\text{Dup1 Result} + \text{Dup2 Result}} \right) \times 200$$

Equation 13. Calculation of dilution factor.

$$\text{Dilution factor} = \frac{\text{Volume of original sample} + \text{Volume of water}}{\text{Volume of original sample}}$$

Equation 14. Mid-to-high level soil sample concentration.

$$\text{Sample Conc.} \left(\frac{\mu\text{g}}{\text{Kg}} \right) = \frac{\text{Raw concentration} \left(\frac{\mu\text{g}}{\text{L}} \right) \times \text{Water volume (L)} \times \left(\frac{\text{Total methanol volume (mL)}}{\text{Methanol volume used (mL)}} \right)}{\text{Sample Weight (Kg)} \times \left(\frac{\% \text{ solids}}{100} \right)}$$

16 ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

- 16.1 DoD Quality Systems Manual supersedes all requirements. For additional information on DoD based projects reference SOP PI-001.
- 16.2 Method Blank. No target analytes may be present in the method blank above the method reporting limit. For DoD projects no analytes may be detected greater than 1/2 the MRL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). The blank result must not otherwise affect the sample results. For common laboratory contaminants, no analytes may be detected greater than the MRL (see Box D-1 of the DoD manual).
- 16.3 Laboratory Control Sample (LCS). Recovery must be within established control limits maintained in LIMS, which may not exceed established method criteria. For LCS duplicates, where prepared and analyzed, RPDs must within established control limits maintained in LIMS.
- 16.4 Matrix spike/matrix spike duplicate (MS/MSD). Recovery and RPD must be within established control limits maintained in LIMS.
- 16.5 Surrogate recoveries. All QC samples (BLK, LCS) must have surrogate recoveries within control limits maintained in LIMS. Other project specific requirements may apply.
- 16.6 Internal standard elution and recoveries.
 - 16.6.1 Internal standard response in each sample and QC sample should be within the limits (shown in **Table 4**) of the response in the reference initial calibration standard (typically equivalent to mid-point in curve).
 - 16.6.2 The retention time of internal standards may not vary by more than 0.5 minutes from the reference initial calibration standard.



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17 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Calibration procedures must follow the requirements of policy QA-008, "Calibration requirements". Corrective actions for most common out-of-control data situations are described in SOP QAQC-32, "Corrective actions for out-of-control data".

- 17.1 BFB Tune: BFB tune criteria must be met before any samples can be reported. The prescribed corrective action is to retune the instrument or perform necessary maintenance.
- 17.2 Initial calibration: Initial calibration and SCV criteria must be met before any samples can be reported. The prescribed corrective action is to re-calibrate.
- 17.3 Continuing calibration:
 - 17.3.1 If the CCV is out of control by two times or more the calibration requirement indicated in this procedure, sample results may not be reported except under extenuating circumstances documented in an NCM.
 - 17.3.2 If the CCV is out of control, it can be reanalyzed once to verify that the analysis was not defective.
 - 17.3.3 Follow the flowchart associated to CCVs in SOP QAQC-32 for additional corrective actions or qualifiers.
- 17.4 Method Blank.
 - 17.4.1 The prescribed corrective action is reanalysis of the method blank to confirm the contamination, which if confirmed, requires re-extraction and analysis of all associated samples.
 - 17.4.2 If there are positive results in the method blank below the reporting limit, qualify data as indicated in the method blank flowchart of SOP QAQC-32.
- 17.5 Laboratory Control Sample (LCS).
 - 17.5.1 The prescribed corrective action is reanalysis of the LCS, which if confirmed, requires re-extraction and analysis of all associated samples.
 - 17.5.2 If LCS indicates high or low bias in the sample results, qualify data as indicated in the method LCS flowchart of SOP QAQC-32.
- 17.6 Matrix spike/matrix spike duplicate.
 - 17.6.1 If recovery exceeds limits established in LIMS, but LCS recovery is in control, matrix effects are possible, if not likely. Non-conforming results must be flagged with LIMS qualifier QM-07 ("The spike recovery was outside acceptance limits for the MS and/or MSD. The batch was accepted based on acceptable LCS recovery.").
 - 17.6.2 If RPD exceeds limits established in LIMS, matrix effects can be ascribed. Non-conforming results must be flagged with LIMS qualifier QM-11 ("Precision between duplicate matrix spikes of the same sample was outside acceptance limits.").
- 17.7 Surrogates



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17.7.1 The prescribed corrective action is re-analysis of the affected samples, unless objective evidence of suspected or confirmed matrix effects is available.

17.7.2 The surrogate flow chart in SOP QAQC-32 shows corrective actions and/or qualifiers that can be applied to surrogate results.

17.8 Internal standards

17.8.1 Failing to meet internal standard recovery requirements may be indicative of a poor run or matrix interferences, and the analyst should use professional judgment in deciding whether the sample requires reanalysis. All actions must be documented in an NCM.

17.8.2 If the retention time of any internal standard in any sample varies by more than 0.5 minute from the reference initial calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

17.8.3 Samples with internal standard recoveries, or retention times, not meeting criteria must be reanalyzed unless there is objective evidence of matrix interferences.

18 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Out-of-Control Data must be qualified as estimated and discussed with project management. Decisions on re-sampling must involve client input. These decisions will be made on a case-by-case basis. All instances of out of control or unacceptable data must be documented in a non-conformance memo (NCM).

19 METHOD PERFORMANCE

19.1 Preparation and analysis of method blanks and laboratory control samples with each batch of up to 20 samples.

19.2 Method detection limit (MDL) studies are performed annually or verified quarterly.

19.3 An initial precision and accuracy study shall be performed before analysis is conducted.

19.4 Continuing demonstrations of precision and accuracy are documented annually for each analyst using results from four LCSs or from successful PT studies. Continuing demonstration results are included in the training files of each analyst.

19.5 Use of control charts to evaluate trends in the performance of the methods.

20 POLLUTION PREVENTION

All hazardous waste shall be disposed of in accordance with all local, state and federal regulations.

21 WASTE MANAGEMENT

21.1 Samples are retained in the laboratory for 45 days after sampling before disposal.

21.2 DOD samples are retained for 60 days after sampling before disposal.

21.3 Samples are disposed of as directed by ENCO SOP ADMIN 29.



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22 REFERENCES

- 22.1 Environmental Conservation Laboratories, Inc., Quality Systems Manual.
- 22.2 ENCO Policy QA-008, "Calibration requirements"
- 22.3 ENCO SOP QAQC-32, "Corrective actions for out-of-control data"
- 22.4 ENCO Policy QA-001, "Sample batches"
- 22.5 ENCO Policy QA-011, "Manual integration"
- 22.6 ENCO Policy QA-006, "Data recording"
- 22.7 U.S. EPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, EPA 814B-92-002, Sept. 92
- 22.8 EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Waste, EPA 624, 1983.
- 22.9 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA 8260B (Rev 2), Update 3, 1996.
- 22.10 Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992.
- 22.11 ENCO SOP ADMIN 29, "Sample Segregation and Disposal"
- 22.12 ENCO Policy QA-013, "Detection Limits"
- 22.13 ENCO Policy QA-012, "Data review"
- 22.14 ENCO SOP QAQC-33, "Training Files"
- 22.15 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1, April 2009
- 22.16 SW-846, Method 8000B Determinative Chromatographic Separations (Rev 2), December 1996

23 MISCELLANEOUS

23.1 Revision Summary

Rev. No	Date Revised	Revision Summary
1		SOP was reformatted and updated.

23.2 Method modifications, enhancements and clarifications

- 23.2.1 All analytes in a CCV are evaluated against the initial calibration curve, and must have a recovery equal to $\pm 20\%$ of the spiked amount. This requirement supersedes the CCC criteria for CCV evaluation referenced in SW846 8260B.
- 23.2.2 EPA 8260B and EPA 624 discuss direct injection techniques. This SOP does not cover any direct injection procedures. Direct injections are not performed.
- 23.2.3 The laboratory does not add sodium thiosulfate to VOA samples as described in section 9.1 of EPA 624. The laboratory checks each container that has been preserved with Sodium Thiosulfate for presence or absence of free and total chlorine. The presence of free or total chlorine in samples should be documented on the preparation bench sheet and in an NCM



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Table 1. Method reporting limits.

Compound	Water (ug/L)	Soil (mg/kg)
1,4-Dioxane	1.3	0.0012

Table 2. Initial calibration curve (concentrations in ug/mL).

Analyte	CAL1 ¹	CAL2	CAL3 ²	CAL4	CAL5
1,4-Dioxane	2	4	25	40	80
Surrogates					
Toluene-d8	1	3	5	8	10
Internal Standards					
1,4-Difluorobenzene	5	5	5	5	5
1,4-Dioxane-d8	50	50	50	50	50

Table 3. Analysis sequence.

Run #	Description	Comment
1	BFB-50 ng (on column)	Used to verify mass spectrometer is properly tuned.
2	Instrument blank	Used to verify instrument cleanliness prior to performing calibration.
3	CAL1	Initial Calibration Curve (if established, skip to No. 10)
4	CAL2	
5	CAL3	
6	CAL4	
7	CAL5	
8	CAL6	
9	CAL7	
10	SCV1 -OR- CCV1	Second-source calibration verification standard if curve was analyzed -OR- Continuing calibration standard to verify continued validity of curve
11-	Samples and QC Samples	As many analyses as can be analyzed within 12:00:00 hours of the acquisition time for BFB (Run No. 1)

¹ This calibration level is analyzed to ensure that the MRL is consistent with regulatory requirements.

² This calibration level is the CCV level.



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Table 4. Calibration and QC requirements.

Calibration or QC check	EPA 8260C
BFB Frequency	12 hours
BFB Tune	Table 5
Initial calibration curve	Five-point curve minimum
Initial calibration curve RSD	15%
Regression curve COD	0.99 ³
Second-source calibration verification	80-120%R
Continuing calibration verification	80-120%R
Method blank	5%, <MRL
LCS	5%, LIMS control limits ⁴
Matrix spikes	5% MS/MSD, LIMS control limits ⁴
Surrogates	LIMS control limits ⁴
Internal standards – RT	Within 30 sec of ICAL reference standard
Internal standards - %R	50-200% of ICAL reference standard

Table 5. BFB ion abundance 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

³ The Department of Defense (DOD) Quality Systems Manual (QSM) requires a coefficient of determination of 0.995.

⁴ Project or program specific criteria may apply – (DOD). South Carolina requires that LCS be within the range of 70-130%, except of poor purgers which have a range of 60-40%.



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Table 6. SIM Mode ion dwell times⁵

Group	Ion	Dwell time in msec
1 (10-12.15 min)	63	100
1 (10-12.15 min)	88	100
1 (10-12.15 min)	114	100
2 (12.15-12.75 min)	58	100
2 (12.15-12.75 min)	88	300
2 (12.15-12.75 min)	64	100
2 (12.15-12.75 min)	96	300
3 (12.75 -14 min)	70	100
3 (12.75 -14 min)	98	100
3 (12.75 -14 min)	100	100

Table 7. SIM Mode temperature program.⁵

Level	Rate (C°/min)	Final Temperature (C°)	Final Time (min)
1	10	50	0
2	15	100	0.50
3	20	200	9

⁵ Retention times, temperatures, dwell times, and ions are subject to change based on instrument performance.



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PREPARATION BENCH SHEET

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Analysis
8260C SIM ID

0G25005
ENCO Orlando

<u>Surrogate Solution</u>	
A0G0034	8260/iso surr toluene d8 (virtual)
<u>Spiking Solution</u>	
A0C0739	8260 1,4-dioxane 2nd working

Prepared using: EPA 5030B_MS

Matrix: Water

Lab Number	Code	Sample ID, Source and Sample Location	Due Date	Analysis	pH adj	Res Cl ⁽¹⁾	Initial (mL)	Final (mL)	Spike ID	ul Spike	ul Surr1	ul Surr2	Extraction Comments
0G25005-BLK1		Blank		QC							1		
0G25005-BS1		LCS		QC					A0C0739	7.5	1		
0G25005-MS1		Matrix Spike [A003790-12]		QC					A0C0739	7.5	1		
0G25005-MSD1		Matrix Spike Dup [A003790-12]		QC					A0C0739	7.5	1		
A003790-12		GR-MW06C.GW34 A[MS33	30-Jul-10	8260C SIM ID							1		1,4-DIOXANE

(1) Before solvent extraction proceeds, verify that there is no residual chlorine above 0.5 mg/L. Any sample above this must be treated to remove excess chlorine before extraction, and this documented as a comment.

Inst 1 SIM ID

Start Date/Time _____	Standard ID# _____	Description _____	Manufacture Lot# _____
Stop Date/Time _____			

Equipment Used			
Sonicator _____	Tuned per manufacturer instructions? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA		
Turbovap _____	Temperature: _____	N2 pressure (initial): _____	(final): _____
Turbovap _____	Temperature: _____	N2 pressure (initial): _____	(final): _____
Turbovap _____	Temperature: _____	N2 pressure (initial): _____	(final): _____
Balance _____	Daily calibration complete? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA		
Other _____			

Samples Prepared By _____ Date _____ Samples Prepared By _____ Date _____ Samples Prepared By _____ Date _____

Figure 1. VOA sample preparation benchsheet.

