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NAS JACKSONVILLE  
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FINAL SAMPLING AND ANALYSIS PLAN FOR REMEDIAL INVESTIGATION FEASIBILITY  
STUDY ADDENDUM FOR OPERABLE UNIT 3 (OU 3) NAS JACKSONVILLE FL  
5/1/2010  
TETRA TECH

# Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



Rev. 1  
05/28/10

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

### Remedial Investigation/Feasibility Study Addendum for Operable Unit 3

Naval Air Station Jacksonville  
Jacksonville, Florida

Contract Task Order 0154

May 2010



NAS Jacksonville  
Jacksonville, Florida 32212-0030

**Project-Specific Sampling and Analysis Plan**  
**Site Name/Project Name:** OU 3, NAS Jacksonville  
**Site Location:** Jacksonville, Florida

**Title:** RI/FS Addendum for OU 3  
**Revision Number:** 1  
**Revision Date:** May 2010

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

**SAMPLING AND ANALYSIS PLAN**  
**(Field Sampling Plan and Quality Assurance Project Plan)**  
**April 2010**

**Remedial Investigation and Feasibility Study Addendum**  
**Operable Unit (OU) 3**  
**Naval Air Station (NAS) Jacksonville**  
**Jacksonville, Florida**

**Prepared for:**  
**Naval Facilities Engineering Command**  
**Southeast**  
**NAS Jacksonville Building 903**  
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**Prepared by:**  
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**Prepared under:**  
**Comprehensive Long-Term Environmental Action Navy**  
**CLEAN Contract No. N62467-04-D-0055**  
**Contract Task Order 0030**

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(UFP-QAPP Manual Section 2.1)

**Document Title:** Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), April 2010, Remedial Investigation and Feasibility Study Addendum, Operable Unit (OU) 3, Naval Air Station (NAS) Jacksonville, Florida

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**SAP Worksheet #1 -- Approval Page**  
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**Lead Organization:** Naval Facilities Engineering Command Southeast

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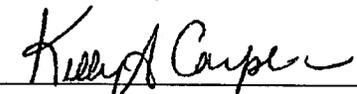
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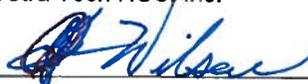
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Peter Dao  
United States Environmental Protection Agency  
Region 4

BUREAU OF WASTE CLEANUP  
RECEIVED

APR 23 2010

FEDERAL PROGRAMS SECTION

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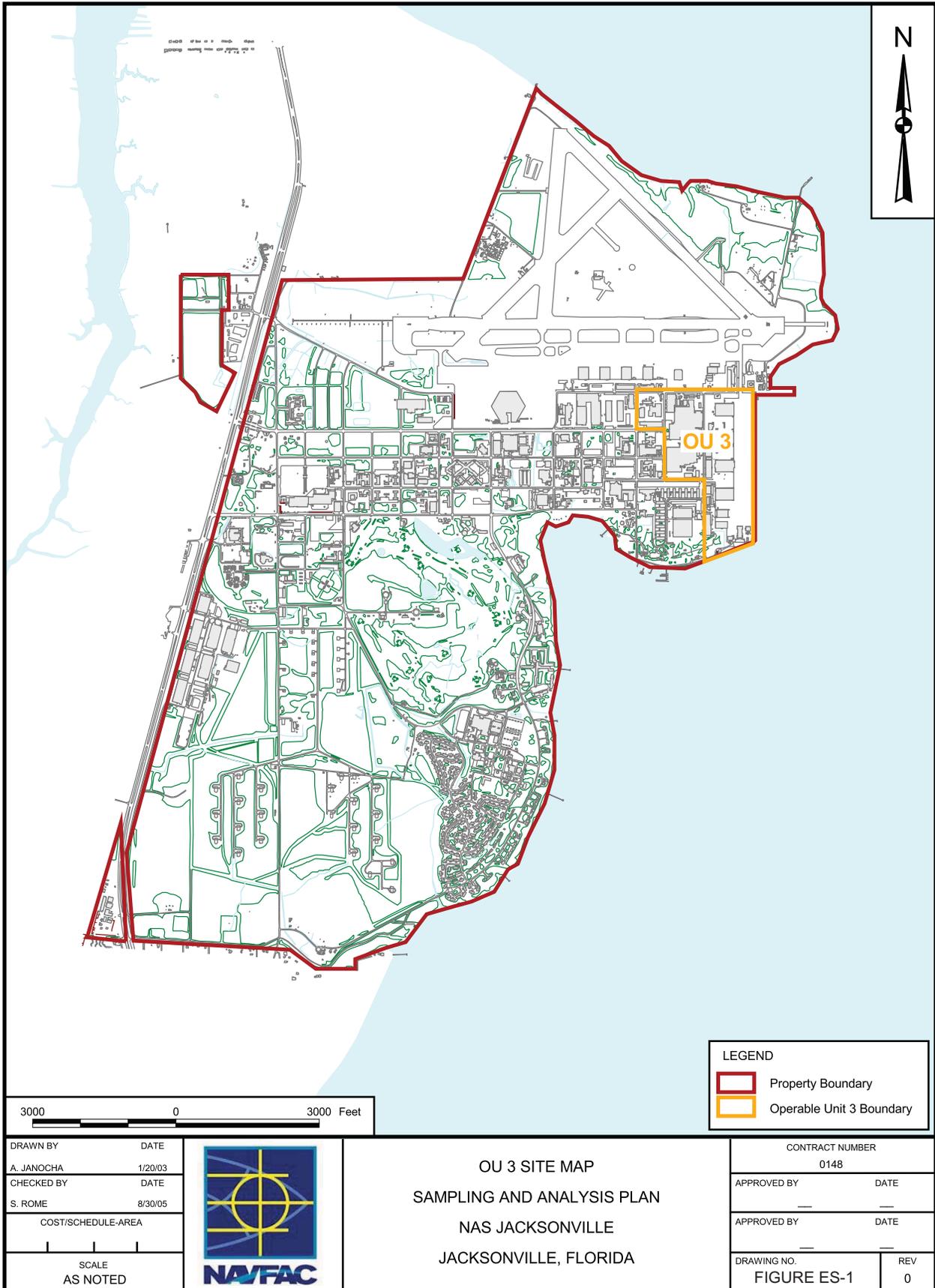
## **EXECUTIVE SUMMARY**

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract No. N62467-04-D-0055, Contract Task Order (CTO) 0154. This plan was prepared for surface soil, groundwater, storm sewer, and surface water/pore water sampling events associated with completion of a Remedial Investigation and Feasibility Study (RI/FS) Addendum for Operable Unit (OU) 3. OU 3 roughly comprises the eastern areas of the installation under the operations of the Fleet Readiness Center Southeast (FRCSE). Figure ES-1 presents a Facility Location Map depicting the location of OU 3.

Since the mid 1990s, several Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)-based investigations, interim actions, and selected remedies have been implemented at OU 3 under the direction of the Naval Air Station (NAS) Jacksonville Partnering Team, which is comprised of representatives of the United States Department of the Navy, the United States Environmental Protection Agency (USEPA), the Florida Department of Environmental Protection (FDEP), the United States Geological Survey (USGS), and Navy contractors. These CERCLA-based actions have addressed Potential Sources of Contamination (PSCs 11-16 and 48) and also areas of groundwater contamination (Areas A through G).

Implementation of post-Record of Decision (ROD) actions has led to optimization of environmental response actions at OU 3. A Five Year Review was conducted in 2005 and, combined with the results of a subsequent optimization study conducted by the United States Department of the Navy (Navy), it was determined by the NAS Jacksonville Partnering Team that additional field actions should be implemented to support an RI/FS Addendum documenting current conditions, and that a UFP-SAP should be prepared. This RI/FS Addendum will then support the development of a new ROD that would address the entirety of environmental issues at OU 3.

This UFP-SAP was generated for, and complies with, applicable Navy, USEPA, and FDEP requirements, regulations, guidance, and technical standards. This includes the Department of Defense (DoD), Department of Energy (DOE), 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 37 standard worksheets specified in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) guidance documents (IDQTF, 2005).



P:\GIS\JACKSONVILLE\_NAS\APRI\OU3\_PCA12.APR SITE LOCATION LAYOUT 3/11/03 AJ

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## ACRONYMS AND ABBREVIATIONS

°C	Degrees Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
ABB-ES	ABB Environmental Services, Inc.
ACL	Alternate Cleanup Limit
AES	Atomic Emission Spectroscopy
Apex	Apex Environmental Engineering & Compliance
ARAR	Applicable or Relevant and Appropriate Requirement
AST	Aboveground Storage Tank
BFB	Bromofluorobenzene
bgs	Below Ground Surface
CA	Corrective Action
CAS	Chemical Abstract Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CH2MHill	CH2M Hill Constructors, Inc.
CLEAN	Comprehensive Long-Term Environmental Action Navy
COC	Contaminant of Concern
COPC	Contaminant of Potential Concern
CPT	Cone Penetrometer Testing
CSM	Conceptual Site Model
CTL	Cleanup Target Level
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Absorption
CVOC	Chlorinated Volatile Organic Compound
CWA	Clean Water Act
DCA	Dichloroethane
DCE	Dichloroethene
DFTPP	Decafluorotriphenylphosphine
DI	De-ionized
DNAPL	Dense Non-Aqueous Phase Liquid

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

DO	Dissolved Oxygen
DoD	Department of Defense
DOE	Department of Energy
DOT	Department of Transportation
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EECA	Engineering Evaluation and Cost Analysis
ELAP	Environmental Laboratory Accreditation Program
Empirical	Empirical Laboratories, LLC
ERA	Ecological Risk Assessment
Ext	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FDER	Florida Department of Environmental Regulation
FID	Flame Ionization Detector
FOL	Field Operations Leader
FRCSE	Fleet Readiness Center Southeast
FS	Feasibility Study
FTMR	Field Task Modification Request
ft/yr	Feet Per Year
G&M	Geraghty & Miller
GC/MS	Gas Chromatograph/Mass Spectrometer
GCTL	Groundwater Cleanup Target Level
Hart	Fred C. Hart Associates, Inc.
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HHRA	Human Health Risk Assessment
HLA	Harding Lawson and Associates
HNO <sub>3</sub>	Nitric Acid
HRC	Hydrogen Releasing Compound
HSM	Health and Safety Manager

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

H <sub>2</sub> SO <sub>4</sub>	Sulfuric Acid
IAS	Initial Assessment Study
IC	Ion Chromatography
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
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
KB Labs	KB Labs, Inc.
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
LUC	Land Use Control
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
mg/kg	Milligrams per Kilogram
MIP	Membrane Interface Probe
mL	Milliliter
MNA	Monitored Natural Attenuation
MPC	Measurement Performance Criterion
MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	Mean Sea Level
NA	Not Applicable
NaOH	Sodium Hydroxide

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast
Navy	United States Department of the Navy
NELAP	National Environmental Laboratory Accreditation Program
NPDES	National Pollutant Discharge Elimination System
NTCRA	Non-Time Critical Remedial Action
NTU	Nephelometric Turbidity Unit
ORP	Oxidation-Reduction Potential
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
OU	Operable Unit
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PCE	Tetrachloroethene
pCi/L	Picocuries Per Liter
PDF	Portable Document Format
PM	Project Manager
PoC	Point of Contact
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit
PQO	Project Quality Objective
PSC	Potential Source of Contamination
PT	Proficiency Testing (previously known as performance evaluation sample)
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
r	Linear Regression Correlation Coefficient
r <sup>2</sup>	Coefficient of Determination
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSL	Regional Screening Level
RT	Retention Time
SAP	Sampling and Analysis Plan
SCTL	Soil Cleanup Target Level
SDG	Sample Delivery Group
SI	Site Inspection
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SPP	Systematic Planning Process
SQL	Structured Query Language
SSFP	Scoping Study Field Program
SSO	Site Safety Officer
SVE	Soil Vapor Extraction
SVOC	Semivolatile Organic Compound
SWCTL	Surface Water Cleanup Target Level
TAL	Target Analyte List
TBD	To Be Determined
TCA	Trichloroethane
TCE	Trichloroethene
TCL	Target Compound List
Tetra Tech	Tetra Tech NUS, Inc.
TOC	Total Organic Carbon
UCL	Upper Confidence Limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
µg/L	Micrograms per Liter
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
VC	Vinyl Chloride
VOC	Volatile Organic Compound

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

**Site Name/Number:** Naval Air Station (NAS) Jacksonville, Florida  
**Operable Units:** Operable Unit (OU) 3  
**Contractor Name:** Tetra Tech NUS, Inc. (Tetra Tech)  
**Contract Number:** N62467-04-D-0055  
**Contract Title:** Comprehensive Long-Term Environmental Action Navy (CLEAN)  
**Work Assignment Number:** Contract Task Order (CTO) 0154

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)* (USEPA, 2005) and United States Environmental Protection Agency (USEPA) *Guidance for Quality Assurance Project Plans, QA/G-5, QAMS* (USEPA, 2002).
2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA); and Resource Conservation and Recovery Act (RCRA).
3. This SAP is a project-specific SAP.
4. List dates of scoping sessions that were held:

Scoping Session	Date
Data Quality Objectives (DQOs) Conference Call	February 20, 2007
DQOs Conference Call	March 26, 2007
DQOs Conference Call	April 28, 2009
DQO Meeting	September 15, 2009
DQO Meeting	February 10, 2010

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

Title	Date
SAP for Additional Groundwater Assessment for OU 3, Area G	August 23, 2004
SAP for Additional Assessment at Buildings 106 and 780 (Tetra Tech)	December 2, 2005
SAP Addendum, Buildings 106 and 780 (Tetra Tech)	July 18, 2006
Sediment Sampling Work Plan for Potential Source of Contamination (PSC) 16, OU 3 (Tetra Tech)	February 20, 2007
SAP for Additional Assessment of Area C, St. Johns River Sampling (Tetra Tech)	January 3, 2008
SAP for Groundwater Assessment Upgradient of Building 106, OU 3	October 31, 2008

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

Florida Department of Environmental Protection (FDEP) (regulatory stakeholder),

USEPA Region 4 (regulatory stakeholder),

NAS Jacksonville (property owner),

7. Lead organization: Naval Facilities Engineering Command Southeast (NAVFAC SE)

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

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
Adrienne Wilson	Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC IPT South Atlantic Code OPA6, Cube 36 135 Ajax Street Jacksonville, FL 32212-0030	(904) 542-6160	Adrienne.Wilson@navy.mil
Tim Curtin	Installation Restoration Program (IRP) Manager/ NAS Jacksonville Point of Contact (PoC)	NAS Jacksonville Building 1, Code 064TC NASJAX /Yorktown/Langley Jacksonville, FL 32212	(904) 542-4228	Tim.L.Curtin@navy.mil
To Be Determined (TBD)	NAVFAC Quality Assurance Officer (QAO)/ Navy Chemist	TBD	TBD	TBD
TBD	Head of Reference Desk (NAS Jacksonville Administrative Record)	TBD	TBD	TBD
David Grabka	RPM/ Provides Regulator Input	Florida Department of Environmental Protection 2600 Blair Stone Road, MS 4535 Tallahassee, FL 32399-2400	(850) 245-8997	david.grabka@dep.state.fl.us
Peter Dao	RPM/ Provides Regulator Input	USEPA Region 4 Atlanta Federal Center 61 Forsyth Street, SW Atlanta, GA 30303-8960	(404) 562-8508	dao.peter@epa.gov
Debra Humbert (copy of cover letter only)	Tetra Tech Program Manager / Manages Navy Initiatives	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-1990	debra.humbert@tetrattech.com

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Todd Romero (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	KB Labs, Inc. (KB Labs) 25132 SW 1st Ave Newberry, FL 32669	(352) 472-5830	toddr@kbmobilelabs.com
Anita Biernacki (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Microbial Insights 2340 Stock Creek Boulevard Rockford, TN 37853-3044	(865) 573-8188 ext. 108	abiernacki@microbe.com
TBD (electronic copy only)	Well Installation Subcontractor PM/ Provides Membrane Interface Probe (MIP) and Direct Push Technology (DPT) Drilling Services	TBD	TBD	TBD

Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of this SAP to all Tetra Tech personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet).

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

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

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

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

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

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
<b>Navy and Regulator Project Team Personnel</b>					
Adrienne Wilson	Navy/ RPM/ Manages Project Activities for the Navy	(904) 542-6160	See Worksheet #1 for signature	All	
Tim Curtin	Navy/ IRP Manager/ NAS Jacksonville PoC	(904) 542-4228		All	

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
David Grabka	FDEP/ RPM/ Provides Regulator Input	(850) 245-8997	See Worksheet #1 for signature	All	
Peter Dao	USEPA Region 4/ RPM/ Provides Regulator Input	(404) 562-8508	See Worksheet #1 for signature	TBD	
<b>Tetra Tech Project Team Personnel</b>					
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Donald Hardison	Tetra Tech/ FOL/SSO/ Manages Field Operation and Site Safety Issues	(904) 730-4669 Ext 227		All	
Kelly Carper	Tetra Tech/ QAM/ Manages NAVFAC SE Contract QA Program and Implementation	(412) 921-7273	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	
Peggy Churchill	Tetra Tech/ Environmental Scientist/ Provides DQO and SAP Support	(321) 636-6470		All	
Mark Traxler	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	(610) 382-1171		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
Lee Leck	Tetra Tech/ Data Manager/ Manages Databases	(412) 921-8856		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
<b>Subcontractor Personnel</b>					
Kim Kostzer	Empirical/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(615) 345-1115		Worksheets #6, #12, #14, #15, #19, #23- 28, #30, and #34-36	
Todd Romero	KB Labs/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(352) 472-5830		Worksheets #6, #15, #19, #23, #24, #25, and #28	
Anita Biernacki	Microbial Insights/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(865) 573-8188, x108		Worksheets #19 and #23	
TBD	TBD/ Subcontractor PM/ Driller for MIP, DPT, and Monitoring Well Installation	TBD		Worksheets # 6, #14, #17, and Figures	

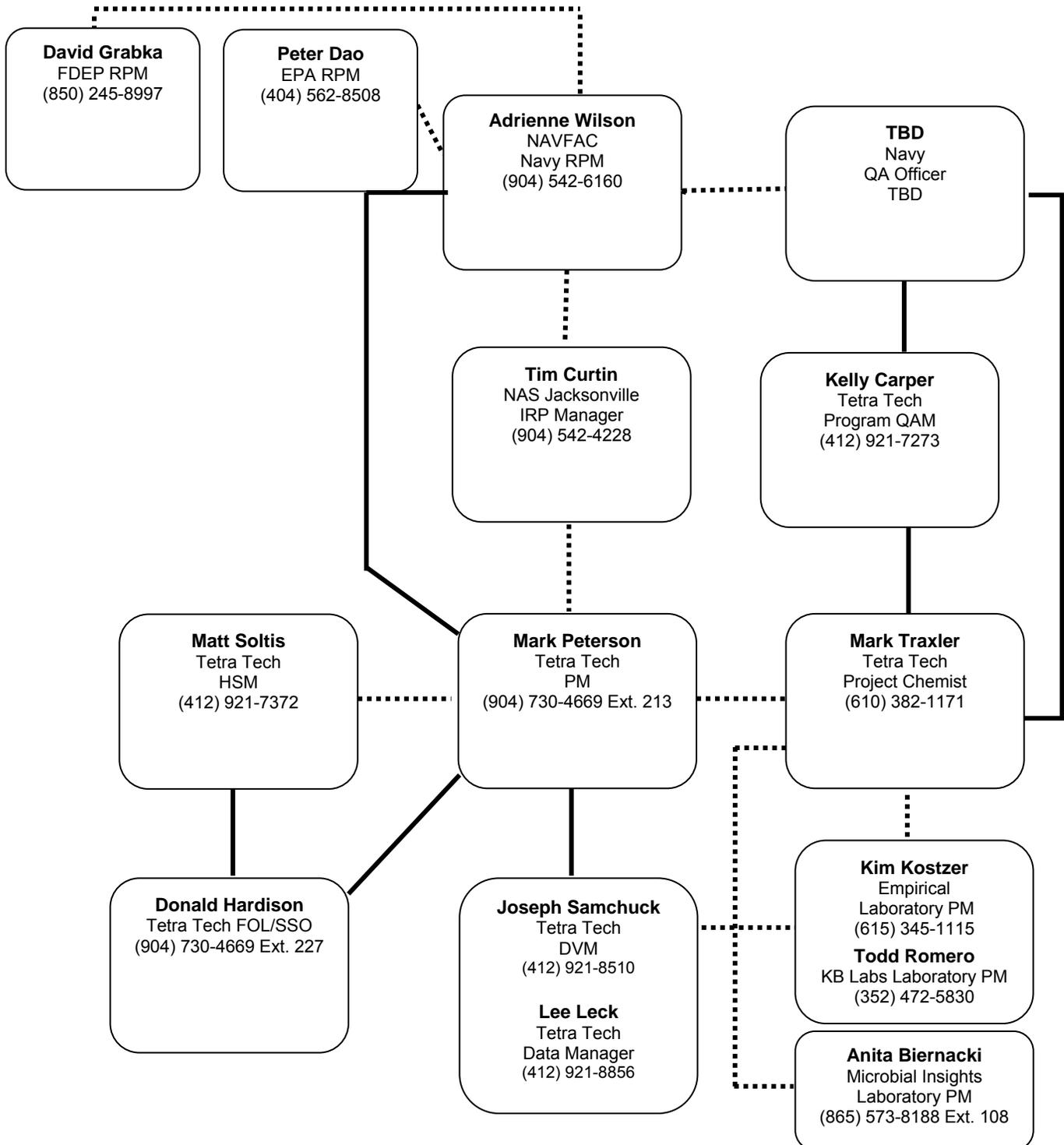
Footnote:

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

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

Lines of Authority —————

..... Lines of Communication



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

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
SAP amendments	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM	Donald Hardison Mark Peterson Adrienne Wilson	(904) 636-6125 Ext 227 (904) 636-6125 Ext 213 (904) 542-6160	<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 five days and send the Navy RPM a concurrence letter within seven 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 of changes to the SAP.</p> <p>The Tetra Tech PM will send scope changes to the Project Team via e-mail within one business day.</p>
Schedule changes	Tetra Tech PM Navy RPM NAS Jacksonville IRP Manager	Mark Peterson Adrienne Wilson Tim Curtin	(904) 730-4669 Ext 213 (904) 542-6160 (904) 542-4228	The Tetra Tech PM will verbally inform the Navy RPM and the NAS Jacksonville IRP Manager on the day that schedule change is known and document via schedule impact letter within one business day of when impact is realized.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM NAS Jacksonville IRP Manager	Donald Hardison Mark Peterson Adrienne Wilson Tim Curtin	(904) 636-6125 Ext 227 (904) 636-6125 Ext 213 (904) 542-6160 (904) 542-4228	<p>The Tetra Tech FOL will verbally inform the Tetra Tech PM on the day the issue is discovered. The Tetra Tech PM will inform the Navy RPM and the NAS Jacksonville IRP Manager (verbally or by e-mail) of the issue within one day of the 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 also send a concurrence letter to the Navy RPM within seven days, if project scope is affected. The Navy RPM will sign the letter within five days of receipt. The Tetra Tech PM will document the change(s) via an FTMR form within two days of identifying the need for change and will obtain required approvals within five days of initiating the form.</p>

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
<p>Stop work recommendations, for example, to protect workers from unsafe conditions/ situations or to prevent a degradation in quality of work/ and initiate work upon corrective action</p>	<p>Tetra Tech FOL/SSO  Tetra Tech PM  Tetra Tech QAM  Navy RPM  NAS Jacksonville IRP Manager</p>	<p>Donald Hardison  Mark Peterson  Kelly Carper  Adrienne Wilson  Tim Curtin</p>	<p>(904) 636-6125 Ext 227  (904) 636-6125 Ext 213  (412) 921-7273  (904) 542-6160  (904) 542-4228</p>	<p>If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the NAS Jacksonville IRP Manager, and the identified Project Team members within one 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>
<p>Corrective action for field program</p>	<p>Tetra Tech QAM  Tetra Tech PM  Navy RPM</p>	<p>Kelly Carper  Mark Peterson  Adrienne Wilson</p>	<p>(412) 921-7273  (904) 636-6125 Ext 213  (904) 542-6160</p>	<p>The Tetra Tech QAM will notify the Tetra Tech PM verbally or by e-mail within one business day that the corrective action has been completed.</p> <p>The Tetra Tech PM will then notify the Navy RPM within one business day (verbally or by e-mail).</p>
<p>Field data quality issues</p>	<p>Tetra Tech FOL/SSO  Tetra Tech PM</p>	<p>Donald Hardison  Mark Peterson</p>	<p>(904) 636-6125 Ext 227  (904) 636-6125 Ext 213</p>	<p>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.</p>

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Laboratory data quality issues	Empirical Laboratory PM KB Labs Laboratory PM Microbial Insights Laboratory PM Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Kim Kostzer Todd Romero Anita Biernacki Mark Traxler Mark Peterson Adrienne Wilson	(615) 345-1115 (352) 472-5830 (865) 573-8188 ext. 108 (610) 382-1171 (904) 636-6125 Ext 213 (904) 542-6160	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered.  The Tetra Tech Project Chemist will notify (verbally or via e-mail) the Tetra Tech PM within one business day. The Tetra Tech PM will notify the Navy RPM within 7 days by letter.

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

Name	Title/Role	Organizational Affiliation	Responsibilities
Adrienne Wilson	RPM/ Manages project	NAVFAC SE	Oversees project implementation, including scoping, data review, and evaluation.
Tim Curtin	NAS Jacksonville PoC – IRP Manager/ Manages daily site activities related to this project	NAVFACE SE NAS Jacksonville	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
Dave Grabka	RPM/ Manages project	FDEP	Participates in scoping, data review, evaluation, and approves the SAP.
Peter Dao	RPM/ Manages project	USEPA Region 4	Participates in scoping, data review, and evaluation.
Mark Peterson	PM/ Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project.
Donald Hardison	FOL/SSO/ Manages field operation and site safety issues	Tetra Tech	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 site-specific HASP.
Kelly Carper	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.

Name	Title/Role	Organizational Affiliation	Responsibilities
Matt Soltis	HSM/ Oversees health and safety activities	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety Program.
Mark Traxler	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.
Joseph Samchuck	DVM/ Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including: <ul style="list-style-type: none"> <li>• Ensures QA of data validation deliverables.</li> <li>• Provides technical advice on data usability.</li> <li>• Coordinates and maintains data validation review schedule.</li> </ul>
Lee Leck	Data Manager/ Manages databases	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.
TBD	Well Installation Subcontractor PM/ Driller for MIP, DPT, and Monitoring Well Installation	TBD	Ensures that project specific requirements are communicated to field personnel.
Kim Kostzer Todd Romero Anita Biernacki	Laboratory PM Laboratory PM Laboratory PM/ Manages project	Empirical KB Labs Microbial Insights	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.

**Note:**

In some cases, one person may be designated responsibilities for more than one position. For example, the Tetra Tech 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**  
(UFP-QAPP Manual Section 2.4.4)

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

One exception to this requirement is for surveyors who will not come in contact with contaminated media.

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

Project Name: NAS Jacksonville Projected Date(s) of Sampling: <u>June 2010 through December 2010</u> Project Manager: Mark Peterson		Site Name: OU 3 Site Location: NAS Jacksonville, Florida			
<b>Date of Session:</b> September 15, 2009; December 8-9, 2009; February 9-10, 2010 <b>Scoping Session Purpose:</b> Develop DQOs with the Navy to support UFP-SAP development					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Adrienne Wilson	Navy RPM	NAVFAC SE	(904) 542-6160	Adrienne.Wilson@navy.mil	Navy RPM
Tim Curtin	NAS Jacksonville PoC – IRP Manager	NAVFACE SE NAS Jacksonville	(904) 542-4228	Tim.L.Curtin@navy.mil	IRP Manager
Mike Singletary **	Technical Support	NAVFAC SE	(904) 542-6303	michael.a.singletary@navy.mil	Technical Support
David Grabka	RPM	FDEP	(850) 245-8997	david.grabka@dep.state.fl.us	FDEP RPM
Peter Dao	RPM	USEPA Region 4	(404) 562-8508	dao.peter@epa.gov	USEPA RPM
Mark Peterson	PM	Tetra Tech	(904) 636-6125 Ext 213	mark.peterson@tetrattech.com	PM
Mike Maughon	Technical Support	Tetra Tech	(843) 886-4547	mike.maughon@tetrattech.com	Technical Support
Casey Hudson	PM-RAC Contractor	CH2M Hill Constructors, Inc. (CH2M Hill)	(770) 604-9182 Ext 54172	casey.hudson@ch2m.com	PM
Hal Davis	Geologist	United States Geological Survey (USGS)	(850) 553-3673	hdavis@usgs.gov	Technical Support
Julie Johnson	Administrative Project Assistant III	Tetra Tech	(904) 730-4669 Ext 224	julie.johnson@tetrattech.com	Scribe

Note: \*\*Mike Singletary was not in attendance at the December 8-9 meeting, but was at the September 2009 and February 2010 meetings.

## **9.1 SCOPING MEETINGS SUMMARY**

Various OU 3 site meetings have been held to discuss OU 3 issues. These meetings conducted over 2007 through 2009 have led the Partnering Team to the development of a path forward for OU 3. This strategy calls for an RI Addendum to support the development of a new ROD designed to address the entirety of OU 3.

A scoping meeting for the RI Addendum was held on September 15, 2009. The results of the Sept 15, 2009 scoping meeting led to identification of the inputs into the decision making process for surface soils, groundwater, sediment pore water, surface water, and indoor air. Study boundaries were confirmed and initial work was completed on decision rules for each of the media except indoor air, which will be addressed in a separate or updated SAP at a later date.

Additional team input into the DQO process was completed during the December 8 and 9, 2009 Partnering Team Meeting. During this meeting, the team completed work on the decision rules for each media, which are documented in Worksheet #11.

One exception to the decision criteria established was associated with discharges of contaminated groundwater to storm sewers at OU 3. Based on input from FDEP, it was determined that storm water regulations that prohibit any illicit discharges from entering storm sewers would be an Applicable, Relevant, and Appropriate Requirement (ARAR) that must be considered in the selection of an appropriate remedy for OU 3. The storm water regulation defines illicit discharges as detectable contamination. As a result, the point of compliance for this ARAR will be considered to be at the point of entry into the storm sewer system. Application of this ARAR to the process could significant impact any decisions to be made regarding appropriate remedies for this pathway.

Subsequent to the September 15 meeting, research conducted by FDEP and USEPA led to an additional discussion of decision rules for contaminated groundwater discharge to storm sewers during the Florida Environmental Alliance Partnering Tier II meeting held on December 1, 2009. During the Tier II discussion, it was determined that the CERLCA process would be allowed to continue to completion and that no immediate enforcement of the storm water prohibition on illicit discharges would be encountered. It was also determined that other potential approaches may be required prior to submittal of the ROD for OU 3 including a potential ARAR waiver, but that no decision would be made at this point in time until additional data are collected and evaluated with remedial alternatives during the Feasibility Study (FS). Attendees at the meeting included the following Florida Partnering Alliance Tier II team members: Sid Allison, Earl Bozeman, Arthur Collins, Jim Crane, Robbie Darby, Jim

**Project-Specific Sampling and Analysis Plan**  
**Site Name/Project Name:** OU 3, NAS Jacksonville  
**Site Location:** Jacksonville, Florida

**Title:** RI/FS Addendum for OU 3  
**Revision Number:** 1  
**Revision Date:** May 2010

Ferro, Helen Lockard, Rich May, Eric Nuzie, Libby Claggett – Scribe, Gayle Waldron – Facilitator, Guests: Greg Campbell, NAS Pensacola PWE, Melissa Dempsey, TtNUS, Pensacola Tier I, John Schoolfield, NAVFAC SE, Pensacola Tier I, Mike Singletary, NAVFAC SE, Gerry Walker, TtNUS, Pensacola Tier I, Patti Whittemore, NAVFAC SE, Pensacola Tier I.

During the NAS Jacksonville Partnering Team meeting conducted on February 9 and 10, 2010, it was determined that preparation of this UFP-SAP would not include an evaluation of the indoor air exposure pathway. Further evaluation of this pathway will be conducted at a future date and an update of the SAP will be prepared to address this aspect of the RI.

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

**10.1 INTRODUCTION**

NAS Jacksonville was commissioned in October 1940 to provide facilities for pilot training and a Navy Aviation Trades School for ground crewmen. The facility is located in Duval County, Florida on the western bank of the St. Johns River. The facility is approximately 3,800 acres in size and its current mission is to provide facilities and support for the operation and maintenance of naval weapons and aircraft. Support facilities include an airfield for air operations and pilot training, a maintenance depot, a Naval Hospital, a Fleet Industrial Supply Center, a Fleet and Family Support Center, and a recreational facility.

The main portion of NAS Jacksonville is bordered to the north by the Timuquana Country Club, to the east and northeast by the St. Johns River, to the south by a residential area, and to the west by Highway 17, with Westside Regional Park, and commercial developments. The facility is located approximately 24 miles inland from the Atlantic Ocean.

NAS Jacksonville is home to Patrol Squadron Thirty (VP-30), the Navy's largest aviation squadron and the only P-3 Orion Fleet Replacement Squadron that prepares and trains U.S. and foreign pilots, air crew, and maintenance personnel for further operational assignments.

Work in support of the base mission includes fuel storage and transportation systems and the overhaul, intermediate maintenance, and repair of aircraft and engines. Maintenance activities at NAS Jacksonville over the years generated a variety of materials, of which some were disposed of on the base. These include materials resulting from construction activities; municipal solid waste and municipal wastewater treatment plant sludge; and miscellaneous industrial wastes, including waste oils or solvents, paints, and spilled fuels. Current disposal practices are regularly surveyed for conformity to local, state, and federal regulations.

**10.2 PHYSICAL SITE DESCRIPTION**

OU 3 is a 134-acre parcel consisting primarily of paved areas located within a large industrial area of NAS Jacksonville (see Figure 10-1). The location of OU 3 within NAS Jacksonville is illustrated on Figure ES-1. OU 3 is comprised of Potential Source of Contamination (PSC) 11 (Building 101), PSC 12 (the Old Test Cell Building), PSC 13 (the Radium Paint Disposal Pit), PSC 14 (the Battery Shop area), PSC 15 (the Solvent and Paint Sludge Disposal area), PSC 16 (the Black Point Storm Sewer Discharge), PSC 48 (the Station's Dry Cleaners – Building 106), and Building 780 and groundwater contamination areas A through G.

### **10.3 PREVIOUS ENVIRONMENTAL INSPECTIONS AND INVESTIGATIONS, AND REGULATORY STATUS - RECORD OF DECISION**

Prior investigations and actions at OU 3 have included CERCLA based Site Inspections (SIs), Engineering Evaluation and Cost Analysis (EECA), non-time critical removal actions (NTCRAs), Remedial Investigations (RIs), and two Separate Records of Decision (RODs).

These environmental actions were designed to address the various PSC 14 (Battery Shop), PSC 15 (Paint Solvent Disposal Pit), PSC 16 (storm water outfall area), and seven of nine identified chlorinated solvent groundwater plumes (Areas A, B, C, D, E, F, and G, and Buildings 106 and 780). A comprehensive ROD dated September 2000 was approved by the NAS Jacksonville Partnering Team that specified remedies for the majority of these areas. Two areas (Area A and Area G) were not addressed specifically by the September 2000 ROD. The remedies specified for each of the other areas is as follows: PSC 14 – Land Use Controls (LUCs); PSC 15 - excavation and off-site disposal; PSC 16 - tar ball and contaminated sediment removal; Building 106 - air sparging and soil vapor extraction (SVE); Building 780 - groundwater treatment and SVE; Areas B and G - monitored natural attenuation (MNA); Areas C and D - enhanced bioremediation and MNA; and Area F - chemical oxidation.

In September 2006, a second OU 3-related ROD was completed to address Area A. The remedy specified for Area A included MNA and LUCs.

A Five Year Review was conducted and approved in September 2005 (Tetra Tech, 2005). The Five Year Review evaluated the protectiveness of the various remedies specified in the September 2000 ROD. The findings of the Five Year Review indicated that some of the remedies specified were no longer effective. As a result of the Five Year Review and the development of risk based corrective action concepts, the NAS Jacksonville Partnering Team began evaluating the concept of developing a new ROD that would encompass all of the identified contamination areas within OU 3, based on utilizing a holistic evaluation of the entirety of OU 3 and the potential environmental impacts to human receptors (site workers, the potential future resident) and ecological receptors within the St. Johns River.

### **10.4 SITE HISTORY**

Operational history of OU 3 consists mainly of the activities associated with the Fleet Readiness Center Southeast (FRCSE). FRCSE has been the major industrial complex at the facility since its inception in 1940. Past and current land uses at OU 3 remain mostly unchanged since FRCSE became the primary tenant in the 1940s. FRCSE operations consist primarily of performing in-depth rework, repair, and modification of aircraft engines and aeronautical components.

#### **10.4.1 PSC 11 Building 101**

PSC 11, Building 101, is the largest building at NAS Jacksonville, covering an area approximately 1,800 feet by 750 feet within OU 3. Building 101 houses diverse operations such as administrative offices, aircraft parts repair, a machine shop, and airplane hangars. Parts of the floor of the main hangar section of Building 101 are constructed of steel plates over steel beams.

Building 101 includes various locations where hazardous materials for the industrial processes conducted by FRCSE were used or stored. Reportedly, there was unauthorized disposal of waste solvents and other materials below the steel plates of the floor in the jetline hangar area for many years. An estimated 2,000 gallons of solvents could have been disposed of in this manner (approximately 1 gallon per week for 40 years). However; no information concerning specific waste disposal activities of chemicals used in Building 101, such as trichloroethene (TCE) and oils, is available.

A portion of the northern half of Building 101 was identified as a PSC during the Initial Assessment Study (IAS) at NAS Jacksonville (Fred C. Hart Associates, Inc. [Hart], 1983). The IAS report documented two releases of hazardous substances at Building 101: (1) solvents were disposed of beneath the steel floor plates in the hangar area, and (2) a mercury spill occurred in the pump shop in the northeast part of Building 101. Following its initial designation, the boundaries of PSC 11 were enlarged to include the plating shop. PSC 11 is now considered to encompass all of Building 101. The Plating Shop was subsequently decommissioned and closed under the NAS Jacksonville RCRA permit.

PSC 11 is considered to be the source for groundwater contamination identified as Area A. An RI/FS and ROD were completed for Area A in 2005 and Area A is currently in post-ROD MNA.

#### **10.4.2 PSC 12 Old Test Cell Building (Building 101K)**

PSC 12, the Old Test Cell Building (Building 101K), is a one story building approximately 40 feet by 50 feet and is located just east of the former engine testing cells along the east side of Building 101 (Figure 10-1). The building reportedly stored chemicals, waste oil, fuel, and solvents used during the testing of engines. The area around the building is completely paved with asphalt to the east and concrete to the north. Based on a 1939 topographic map of the Building 101K area, approximately 4 to 5 feet of fill has been used to achieve the present elevation. In addition, a previously bermed depression occupied the area. The 1939 map showing this area does not name or indicate the prior use of the bermed feature.

The Old Test Cell Building was identified as a PSC during the IAS because 55-gallon drums of chemicals, such as waste oil, fuel, and solvents were once stored there (Hart, 1983). Numerous spills of toxic and

reactive chemicals from ruptured or rusted drums reportedly occurred at PSC 12. Also, solvents and other wastes were potentially discharged via ruptures and breaks of sanitary and industrial storm sewer interconnections at the building (HLA, 2000a).

#### **10.4.3 PSC 13 Radium Paint Disposal Pit**

PSC 13, Radium Paint Disposal Pit, was located between existing Buildings 840 and 167 in the central part of the FRCSE. The Radium Paint Disposal Pit was identified as a PSC during the IAS because radioactive radium paint waste from the painting of aircraft instrument dials was disposed of in the pit from World War II to the late 1950s (Hart, 1983). The pit was approximately 50 feet long, 40 feet wide, and 1 foot deep when it was active in the 1940s and 1950s. The pit was excavated during the late 1950s, and the soil was moved to PSC 18, the Fill Disposal Area (adjacent to Mulberry Cove).

In 1985, Geraghty & Miller, Inc. (G&M) conducted a verification study at PSC 13 (G&M, 1985); the Verification Study Report concluded that radium-226 only slightly exceeded the Florida Department of Environmental Regulation (FDER) drinking water standard of 5 picocuries per liter (pCi/L). Therefore, the report did not recommend a characterization study at PSC 13.

#### **10.4.4 PSC 14 Battery Shop**

PSC 14, the Battery Shop, was identified as a PSC during the IAS because lead battery acid was disposed of in a seepage pit on the west side of the shop (Hart, 1983). The IAS report estimated that 100 gallons of lead battery acid were disposed of annually from 1959 to 1982. The sink, used to dispose of the battery acid, was taken out of service and disconnected from the seepage pit. The unused pit, consisting of a 30-inch diameter sump approximately 6 feet deep with concrete cover, is still in place. Nickel-cadmium batteries were also stored and used at the Battery Shop.

PSC 14 was included in the 2000 ROD for OU 3. LUCs were implemented to prevent potential exposure to contaminated media.

#### **10.4.5 PSC 15 Solvent and Paint Sludge Disposal Area**

PSC 15, Solvent and Paint Sludge Disposal Area, is located within the FRCSE, was identified as a PSC during the IAS because waste solvents and paint were disposed of from approximately 1968 to 1978 (Hart, 1983). The Solvent and Paint Sludge Disposal Area is an approximately 10,000 square foot area south of the paint shop (Building 868), near the south end of FRCSE (Figure 10-1). The Verification Study report later indicated that solvents and paint were disposed at PSC 15 for 36 years (G&M, 1985). Both reports estimated that 2,000 gallons of waste per year were disposed of at PSC 15.

A removal action was subsequently performed at PSC 15 to remove impacted soils. Some material beneath a storm sewer line and paved areas could not be removed. Groundwater contamination from this area was designated as Area G and was included in the 2000 ROD. Area G is currently in a MNA program.

#### **10.4.6 PSC 16 Black Point Storm Sewer Discharge**

PSC 16 Black Point Storm Sewer Discharge encompasses the outfall of the storm water sewer that drains the southern half of FRCSE. PSC 16 is south of, and adjacent to, OU 3. The Black Point Storm Sewer Discharge to the St. Johns River was identified as a PSC during the IAS based on recurring discharges of JP-5 fuel and oil that reportedly entered the storm sewer from a fuel tank overflow in the vicinity of test cell 12 (Hart, 1983), located along the east side of Building 101 (PSC 11). A spill log from the NAS Jacksonville Facilities Department documented many spills at the Black Point Outfall (PSC 16), including spills of JP-5 fuel, hydraulic oil, chrome, and cyanide (HLA, 2000a). In addition, oil and various chemical wastes from other sources within the southern half of FRCSE were reportedly discharged into the storm sewer.

The storm sewer under FRCSE generally conducts water south along Wright and Wasp Streets and east along Enterprise Avenue to the aircraft apron area. Storm water discharge is then directed south to the St. Johns River at Black Point (Figure 10-1).

A tar ball and contaminated sediment removal action was specified as the remedy for PSC 16 in the 2000 ROD. This work has been completed and subsequent toxicology testing has shown that contaminant concentrations within treatment area sediment are within established background conditions for the St. Johns River (CH2M Hill, 2005).

#### **10.4.7 PSC 48 Building 106**

PSC 48 (Dry Cleaners) Building 106 operated as a dry cleaning facility for the Station from 1962 to 1990. Dry cleaning system configurations consisted of one dry cleaning machine and one post dry cleaning machine. A 150-gallon aboveground storage tank (AST) containing tetrachloroethene (PCE) was located in the southeastern corner of Building 106 (ABB-ES, 1995). The dry cleaning system was upgraded in 1990, and the AST was removed. Dry cleaning operations have since ceased and Building 106 has been razed.

An interim action was conducted to treat groundwater at Building 106. An air sparging and SVE system was operated from 2002 to 2005 when it was discontinued. PSC 48 will be a focus area for the RI Addendum.

#### **10.4.8 Building 780**

Building 780 operated as a paint shop and chemical stripping facility for aircraft and associated parts from 1970 to the mid-1980s. Solvents used during stripping operations consisted of 1,1,1-trichloroethane (1,1,1-TCA); TCE; dichloromethane (methylene chloride); butyl acetate; and naphthalene (ABB-ES, 1995). Spent paints and solvents were also emptied into floor drains and an industrial sewer system. In 1992, Building 780 was converted into a Clean Water Act (CWA) pre-treatment system facility. Building 780 is in current operational use as a solvent recycling facility.

An interim action was conducted to treat groundwater at Building 780. A groundwater extraction and SVE system was operated from 2002 to 2005 when it was discontinued. Building 780 will be a focus area for the RI Addendum.

#### **10.4.9 Area A**

Area A is an area of identified groundwater contamination that is located along the east side of PSC 11 Building 101 with Wright Street running through the site. The area above Area A is flat and consists of either buildings, paved storage areas, or a paved road (Wright Street). Airplane engines and their components were reportedly steam-cleaned in this area. Following steam cleaning operations, the engines were often disassembled and the various parts were cleaned with solvents and other cleaning compounds. The area was reportedly unpaved during the time it was used for cleaning engines. The cleaning system has since been removed and the area has been paved.

Area A is currently in a MNA program required by the 2005 ROD for Area A.

#### **10.4.10 Area B**

Area B consists of an area of groundwater contamination located at the southwest corner of Building 840 (Figure 10-1). During the RI for OU 3, volatile organic compounds (VOCs) were identified at concentrations exceeding FDEP Groundwater Cleanup Target Levels (GCTLs) in the intermediate zone of the surficial aquifer at a depth of 38 feet below ground surface (bgs). Based on a risk evaluation conducted during the RI/FS, additional action was recommended to address the VOCs.

The selected remedy for Area B in the 2000 ROD was MNA.

#### **10.4.11 Area C**

Area C is located between the former location of Hangars 122 and 123. The general location of Area C within OU 3 is depicted on Figure 10-1. Area C was initially identified as an area of elevated groundwater

contamination during a 1993 investigation. No evidence to date identifies the source of the groundwater contamination. Additional sampling and analysis conducted from 1993 to 1998 identified TCE at 5,000 micrograms per liter ( $\mu\text{g/L}$ ) and methylene chloride at 27  $\mu\text{g/L}$  as the predominant contaminants (HLA, 2000a). The plume is estimated to encompass 29,400 square feet, with detected concentrations at depths between 30 and 64 feet bgs (CH2M Hill, 2006).

The selected remedy for Area C in the 2000 ROD was treatment via enhanced bioremediation, which consisted of injection of hydrogen releasing compound (HRC) and post-remediation monitoring.

#### **10.4.12 Area D**

Area D is located on the west end of PSC 11 Building 101 and was discovered as an area of elevated groundwater contamination during a 1993 investigation. No evidence to date identifies the source of groundwater contamination at Area D. Additional sampling and analysis conducted from 1993 to 1998 identified TCE at 6,800  $\mu\text{g/L}$ ; 1,2-dichloroethene (DCE) at 190  $\mu\text{g/L}$ ; PCE at 34  $\mu\text{g/L}$ ; methylene chloride at 11.25  $\mu\text{g/L}$ ; manganese at 662  $\mu\text{g/L}$ ; and arsenic at 23  $\mu\text{g/L}$  as the predominant contaminants (HLA, 2000a). The plume is estimated to encompass 134,050 square feet, with detected concentrations at depths between 27 and 52 feet bgs (CH2M Hill, 2006).

Based on the RI/FS (HLA, 2000a), Area D represented the largest area of contamination at OU 3, including some portions beneath Buildings 103, 101, and 101S.

The selected remedy for Area D in the 2000 ROD was treatment via enhanced bioremediation, which consisted of injection of HRC and post-remediation monitoring.

#### **10.4.13 Area E**

The general location of Area E is at the southern end of the Building 101 hangar area north of Enterprise Avenue. The Scoping Study Field Program (SSFP) by ABB-ES identified groundwater contamination within Area E. The source of the contamination appears to be related to a single discharge/spill event and/or preferential transport from an unidentified upgradient source. Groundwater contamination at Area E consisted primarily of PCE and its daughter products, with a maximum detected concentration of 16,000  $\mu\text{g/L}$  for PCE. Other constituents previously detected in this area were acetone, carbon disulfide, chloroform, and chloromethane.

As reported in the OU 3 RI/FS (HLA, 2000a), the groundwater from Area E appears to be flowing directly toward the storm sewer beneath Enterprise Avenue. The storm sewers in this part of the station

discharge into the St. Johns River. Although this area was assessed in 2004, no further action has been taken at Area E.

#### **10.4.14 Area F**

Area F (MILCON P-615) is located on the east side of Wright Street approximately 600 feet south of the intersection with Enterprise Avenue (Figure 10-1). ABB-ES conducted an investigation during the week of March 9, 1992 that included sampling of shallow soil and groundwater. At the time of the investigation, Area F was the planned location of Waste Treatment Plant Building No. 1. Area F is surrounded by Building 796 to the north, Building 795 to the south, and the Aircraft Final Finish Facility (Building 868) to the east. The 1992 SI was conducted "to evaluate the presence, magnitude, and characteristics of hazardous substances, if any, on the site prior to construction activities" (HLA, 2000a).

The specified remedy for Area F in the 2000 ROD was treatment of TCE and its daughter products with chemical oxidation and post-treatment monitoring. However, during an assessment conducted to design the chemical oxidation treatment, it was found that contamination levels were lower than expected and not suitable for treatment. Subsequently, additional assessment of Area F has redefined the boundaries of the plume and potential impacts to a storm sewer. As a result, Area F will be a focus of the RI Addendum.

#### **10.4.15 Area G**

Area G is near Area F and may be impacted by VOC migration from PSC 15, which is a former solvent and paint sludge disposal area. Radium-226 was identified in shallow soils in this area, resulting in a removal action. Although the removal action was designed to address radium-226, soils impacted with VOCs were also removed.

Due to concerns for structural stability, some impacted soils were left in place beneath utilities and a nearby concrete pad. Subsequent to the removal actions, VOCs were identified at concentrations in excess of GCTLs in groundwater samples collected at Area G. VOC concentrations exceeded GCTLs to depths of 40 feet bgs, with the highest concentrations reported from a depth of approximately 20 to 25 feet bgs (Apex, 2005).

The selected remedy for Area G in the 2000 ROD is periodic monitoring of natural attenuation processes (e.g., biodegradation, dispersion, dilution, sorption, volatilization, chemical or biological stabilization, transformation, or destruction).

## **10.5 CONCEPTUAL SITE MODEL**

Implementation of post-ROD actions has led to efforts to optimize environmental response actions at OU 3. A Five Year Review was conducted in 2005 and, combined with the results of a subsequent optimization study conducted by the Navy, it was determined by the NAS Jacksonville Partnering Team that additional field actions should be implemented to support an RI/FS Addendum documenting current conditions, and that a UFP-SAP should be prepared to document those actions. Based on the direction of the Partnering Team, it was determined that an RI Addendum would be completed to address data gaps that were identified prior to completion of the comprehensive OU 3 ROD. The data gaps included surface soil data from the boundary of OU 3; groundwater data from various areas within or adjacent to OU 3; sediment pore water and surface water from areas potentially receiving recharge from contaminant plumes; water discharging from storm sewers; and information to support an evaluation of the indoor air vapor intrusion pathway. The indoor air vapor intrusion pathway effort will be addressed in a separate or updated SAP at a later date. This RI/FS Addendum will then support the development of a new ROD that will address the entirety of environmental issues at OU 3.

The conceptual site model (CSM) for OU 3 is described in the following sections and depicted on Figures 10-2 through 10-7. Figure 10-2 illustrates the locations of groundwater contamination areas and highlights the widespread nature of TCE contamination. Figure 10-3 provides a plan view of the alignment of CSM cross sections shown on Figures 10-4 and 10-5 for plumes in the northern and southern parts of OU 3, respectively. The CSM presented on Figures 10-4 and 10-5 illustrates the stratigraphy, hydrogeology, primary source areas, contamination migration pathways, and potential receptors. Figure 10-6 shows the locations of storm sewer outfalls and the layout of the storm water sewers in OU 3. Figure 10-7 shows the predictive plume migration pathway and sewer infiltration in Area G.

The purpose of this investigation is to collect additional data to refine the CSM and prepare an RI/FS Addendum for OU 3. As described in the following sections, significant uncertainty remains about contaminants of concern (COCs) potentially discharging to the St. Johns River from groundwater and storm sewers, potential vapor intrusion risk to on-site building occupants, the effectiveness of previous source reduction interim measures and bio-barrier containment pilot tests, the efficacy of natural attenuation processes for COCs in groundwater and river sediments, the nature and extent of source area COCs at depth in the vicinity of Building 106, and the extent to which LUCs should be implemented at OU 3. Based on a Team decision, the soil vapor intrusion pathway will be investigated under a separate effort and thus is not included in this SAP.

Table 10-1 lists the COCs for groundwater at OU 3.

**TABLE 10-1  
 SUMMARY OF GROUNDWATER CONTAMINANTS OF CONCERN FOR OU 3**

Contaminants of Concern	Impacted Site	Regulatory Criterion (µg/L)
<i>cis</i> -1,2-Dichloroethene ( <i>cis</i> -1,2-DCE)	106 and 780	70
<i>trans</i> -1,2-Dichloroethene ( <i>trans</i> -1,2-DCE)	106	100
Total 1,2-Dichloroethene (1,2-DCE)	106 and 780	63
Isopropyl benzene	106	0.8
Tetrachloroethene (PCE)	106 and 780	3
Trichloroethene (TCE)	106 and 780	3
1,1,1-Trichloroethane (1,1,1-TCA)	780	200
1,1-Dichloroethane (1,1-DCA)	780	70
1,1-Dichloroethene (1,1-DCE)	780	7
1,2-Dichloroethane (1,2-DCA)	780	3
Chloroethane	780	12
Toluene	780	40
Vinyl Chloride (VC)	106 and 780	1

µg/L = micrograms per liter  
 Regulatory Criterion = Florida GCTLs

Source: ROD for OU 3 (HLA, 2000b)

### 10.5.1 Geology and Hydrogeology

The stratigraphy at OU 3, which consists of silty to clayey sands interbedded with layers of clay and sandy clay, greatly influences the movement of groundwater and associated contaminants (Figures 10-4 and 10-5). In the northern half of OU 3 (i.e., in the vicinity of Buildings 106 and 780), a clay layer separates the shallow and intermediate zones of the surficial aquifer and may be thinned, discontinuous, or absent in the east-northeast vicinity of Building 106 (USGS, 1998). The characteristics of the clay layer were partly determined by the USGS from hydraulic head measurements and vary from west (Building 106) to east (Building 780), as well as other areas of OU 3 south and outside of the study area of this assessment. The thickness of the clay layer varies from less than 5 feet to 20 feet (USGS, 1998). Near Building 106, the depth to the top of the clay layer ranges from 16 to 18 feet bgs, and from 24 to 26 feet bgs near Building 780 (ABB-ES, 1995). In addition to the clay layer, a thick layer of low-permeability channel-fill deposits bisect the approximate center of OU 3 from northeast to southwest. These channel-fill deposits partially impede the hydraulic connectivity of the northern portion of the intermediate zone from the southern portion of the intermediate zone (USGS, 1998).

Aquifer tests (pumping tests) conducted in the northern (i.e., vicinity of Buildings 106 and 780) and southern portions of OU 3 were used to determine a groundwater migration velocity of approximately 2 feet per year (ft/yr) above the clay layer as compared to about 12 to 35 ft/yr below the clay layer (ABB-ES, 1998). In addition, a downward gradient exists in the center of OU 3 in the immediate vicinity of the channel-fill deposits. This downward gradient appears to be the result of the interruption of groundwater

flow direction from northwest to southeast in the intermediate aquifer (i.e., beneath the clay layer) caused by the low-permeability channel-fill deposits (USGS, 1998).

Groundwater migration above the clay layer in the northern part of OU 3 is also affected by leaky storm sewers with invert located beneath the water table. With groundwater flow constrained by the low permeability soils, the seawall at the St. Johns River, and the underlying clay layer, the groundwater infiltration to the sewers is a significant migration pathway (Figure 10-4).

Below the clay layer in the northern portion of OU 3, groundwater flows east toward the St. Johns River and then upward along the saltwater interface, which results from density differences in the freshwater aquifer and saline groundwater beneath the river. During a barge-mounted DPT investigation conducted as part of the Additional Assessment of Area C, St. Johns River Sampling (Tetra Tech, 2008), a dredged basin was discovered in the river within 200 feet from shore. The dredged basin breached the clay layer in this area and created a preferential flow path for freshwater groundwater to discharge upward through the dredged basin, which had filled over time with organic-rich silty sediment, to the river bottom (Figure 10-4).

In the southern part of OU 3 (i.e., in the vicinity of Areas F and G) the clay layer does not exist (Figure 10-5) and groundwater generally flows to the river along preferential pathways created by storm sewers and the termination of the seawall in the vicinity of Outfall I-6 (Figure 10-6). The groundwater in the southern area then flows upward along the saltwater interface and discharges to the river bottom closer to the shoreline than in the northern part of OU 3 because there is no clay layer or seawall to impede the flow. Groundwater flow in the southern area is affected by infiltration into leaky storm sewers similar to that in the northern area. Results of predictive groundwater modeling performed by USGS in 2009 indicate estimated infiltration rates for the eastern storm sewer in Area G. This infiltration is apparently containing and redirecting the groundwater plume in this area (Figure 10-7).

## **10.5.2 Nature and Extent of Contamination**

### **10.5.2.1 Soils**

Because the vast majority of OU 3 is already covered with buildings and concrete or asphalt pavement there is no direct exposure pathway for potential receptors and soil contamination impacts to groundwater through leaching is effectively limited. In addition, interim measures included removal of contaminated soils in many identified source areas. Because of the size and complexity of OU 3, the NAS Jacksonville Partnering Team has agreed that soils within the boundary of OU 3 will not be a primary focus of the investigation. Since OU 3 will remain industrial in use for the foreseeable future, soil investigations will be

limited to delineation of contamination within the shallow surface soils along the boundaries of OU 3 for the purpose of establishing LUCs.

### **10.5.2.2 Groundwater**

Sources of groundwater contamination are described in Section 10.2. The primary COCs in groundwater are chlorinated solvents. The Five Year Review, subsequent optimization study, and more recent MIP and soil boring data confirmed the large scale of groundwater contamination, with the likelihood of the presence of dense non-aqueous phase liquids (DNAPL), multiple source areas, and complexity of the site with large dilute and comingled plumes. Consequently, the Partnering Team decided to consider OU 3 as a whole and to reconsider the separate RODs at individual areas of groundwater contamination in favor of a more comprehensive OU 3 ROD with primary focus on potential groundwater contamination discharge to the St. Johns River, the receptor most likely to have a completed exposure pathway.

Buildings 106 (PSC 48) and 780 are the primary sources of groundwater contamination in OU 3 as illustrated on Figures 10-2 and 10-4, which are based on monitoring well and DPT sampling data available through April 2009. Figure 10-2 illustrates the widespread extent of TCE contamination in groundwater across OU 3 and potential discharge to the St. Johns River east of Area C and south of Area G. Area F and G source areas and related groundwater contamination (Total VOCs) in the southern part of OU 3 are illustrated on Figure 10-5. Total VOC concentrations and depth of contamination is significantly less in the Area F and Area G plume than in the Buildings 106 and 780 plume.

Area A is also a source of relatively high chlorinated VOCs (CVOCs) concentrations but plume migration is contained by lower permeability soils in this area. The existing ROD for Area A specifies MNA as the remedy.

Cone Penetrometer Testing (CPT) data indicates the clay layer in the northern part of OU 3 varies in thickness from less than 1 foot to approximately 10 feet. MIP and DPT data indicate CVOCs migrated through the thin clay layer at Building 106. PCE concentrations on the order of several thousand ug/L were detected above and below the clay layer to depths of 50-70 feet bgs along the upgradient edge of Building 106.

Total VOCs were measured as high as 44,300 ug/L in groundwater. MIP data indicates much of the saturated zone contaminant mass has diffused into the clay layer and interbedded clay lenses in the surficial zone. This has a significant effect on the duration of remediation for most remedial alternatives, which are generally effective only in higher permeability zones where reagents can be effectively distributed to contaminant zones. As the higher permeability zones are remediated, the slow rate of back

diffusion from the clay and other low permeability zones provides a continuing long-term source to the plume.

For Building 780, the high concentration CVOCs mostly remain above and within the clay layer. The CVOC plume in the surficial zone above the clay layer is significantly constrained by infiltration to the leaky storm sewers, low permeability sandy clay soils, and the seawall.

Interim source reduction measures were previously implemented and then discontinued at Buildings 106 and 780. A bio-barrier containment pilot test was performed at Area C.

Significant uncertainty remains regarding the source of contamination upgradient of Building 106. Contamination in this area could be the result of Building 106 source contaminants migrating through clay layer discontinuities and dispersing down and upgradient along preferential pathways. Alternatively, the upgradient contamination could be due to an unidentified upgradient source. Additional data will be collected in this area to bound the plume and identify the source.

Uncertainty also exists regarding nature and extent of groundwater contamination between the Area C and Area D plumes and at depth below Building 106 where little data has been collected.

### **10.5.3 Migration Pathways**

#### **10.5.3.1 Groundwater to Surface Water**

The potential for groundwater contamination to discharge to the St. Johns River is directly related to the geology and hydrogeology described in Section 10.5.1.

Below the clay layer in the northern part of OU 3 (i.e., in the vicinity of Buildings 106 and 780), groundwater contamination flows east toward the St. Johns River and then upward along the saltwater interface, which results from density differences in the freshwater aquifer and saline groundwater beneath the river. During a barge-mounted DPT investigation conducted as part of the Additional Assessment of Area C, St. Johns River Sampling (Tetra Tech, 2008), a dredged basin was discovered in the river within 200 feet from shore. The dredged area breached the clay layer in this area creating a preferential flow path for freshwater groundwater to discharge through the dredged basin, which has filled over time with organic-rich silty sediment, to the river bottom (Figure 10-4). Pore water samples collected by DPT at ten-foot depth intervals below and within the dredged basin confirms CVOCs are discharging to the dredged basin. However, the migration pathway is not complete because the CVOCs are fully attenuated approximately 15 to 20 feet below the top of the organic-rich silty sediment before actually discharging to

surface water. Additional sampling is planned to confirm the groundwater discharge area and whether contamination discharges to surface water.

In the southern part of OU 3 (i.e., in the vicinity of Areas F and G), the clay layer does not exist (Figure 10-5) and groundwater contamination generally flows toward the river along preferential pathways created by storm sewers and the termination of the seawall in the vicinity of Outfall I-6 (Figure 10-6). The groundwater in the southern area flows upward along the saltwater interface and discharges to the river bottom closer to the shoreline than in the northern part of OU 3 because there is no clay layer or seawall to impede the flow. Significant uncertainty exists as to whether the contamination migration pathway is complete in this area because no pore water data has been collected in the river sediments offshore from Area G. Additional DPT data is planned to be collected along the shoreline to better delineate the plume migration pathway to the river. Pore water sampling in the river sediments is planned to delineate the groundwater discharge area and determine if groundwater contamination is discharging to surface water.

#### **10.5.3.2 Groundwater to Surface Water via Storm Sewer**

Migration of groundwater contamination above the clay layer in the northern part of OU 3 is affected by leaky storm sewers with inverts located beneath the water table. With groundwater flow constrained by the low permeability soils, the seawall at the St. Johns River, and the underlying clay layer, the groundwater infiltration to the sewers is a potentially significant contamination migration pathway (Figure 10-4). Significant uncertainty exists in this area regarding a completed pathway to surface water since no sewer manholes or outfalls have yet been sampled.

Migration of groundwater contamination in the southern area is affected by infiltration into leaky storm sewers similar to that in the northern area. Results of predictive groundwater modeling performed by USGS in 2009 indicates estimated groundwater infiltration rates for the eastern storm sewer in Area G. The infiltration to sewers is apparently containing and helping redirect the groundwater plume in this area (Figure 10-7). Results of sampling performed in 2008 (CH2M Hill, 2008) indicate slight exceedances of Marine Surface Water Cleanup Target Levels (SWCTLs) for vinyl chloride and 1,1-dichloroethene, particularly at low tide, in a grated drain approximately 350 feet from the shoreline. However, Outfall I-6 (Figure 10-6) has not yet been sampled because of difficulty locating it. Facility construction drawings indicate the outfall is submerged in the river approximately 100 feet from shore. Dye testing indicated the integrity of the offshore sewer line is questionable as dye was observed in surface water approximately 25 feet from shore. The facility drawings also show two sewer access locations that are closer to the river but not yet sampled – a manhole within 150 feet of the shoreline and a grated drain within 50 feet. To determine if there is a completed contamination migration pathway to surface water in this area,

additional sampling is planned over the full tidal cycle for the manhole and/or grated drain closest to the shoreline, the sewer outfall, and any identified offshore breaches in the sewer.

Similarly, to confirm whether complete pathways exist at other outfalls in OU 3, sampling of the manhole or drain closest to the shoreline and/or the sewer outfall is planned for all storm sewers located within areas of shallow groundwater contamination and having invert elevations below the water table.

### **10.5.3.3 Vapor Intrusion**

The vapor intrusion pathway to on-site building occupants will be evaluated in a subsequent investigation and will be included in a new or updated UFP-SAP.

### **10.5.4 Potential Receptors**

Potential receptors are site workers, the potential future resident, and aquatic receptors in the St. Johns River. Utility workers have the greatest chance of direct exposure to contaminated soil or groundwater. However, because the site is primarily covered by asphalt and concrete, the likelihood of direct exposure for most receptors is significantly reduced and expected to be managed with LUCs. The groundwater at OU 3 is not used as a drinking water source and is not expected to be in the future.

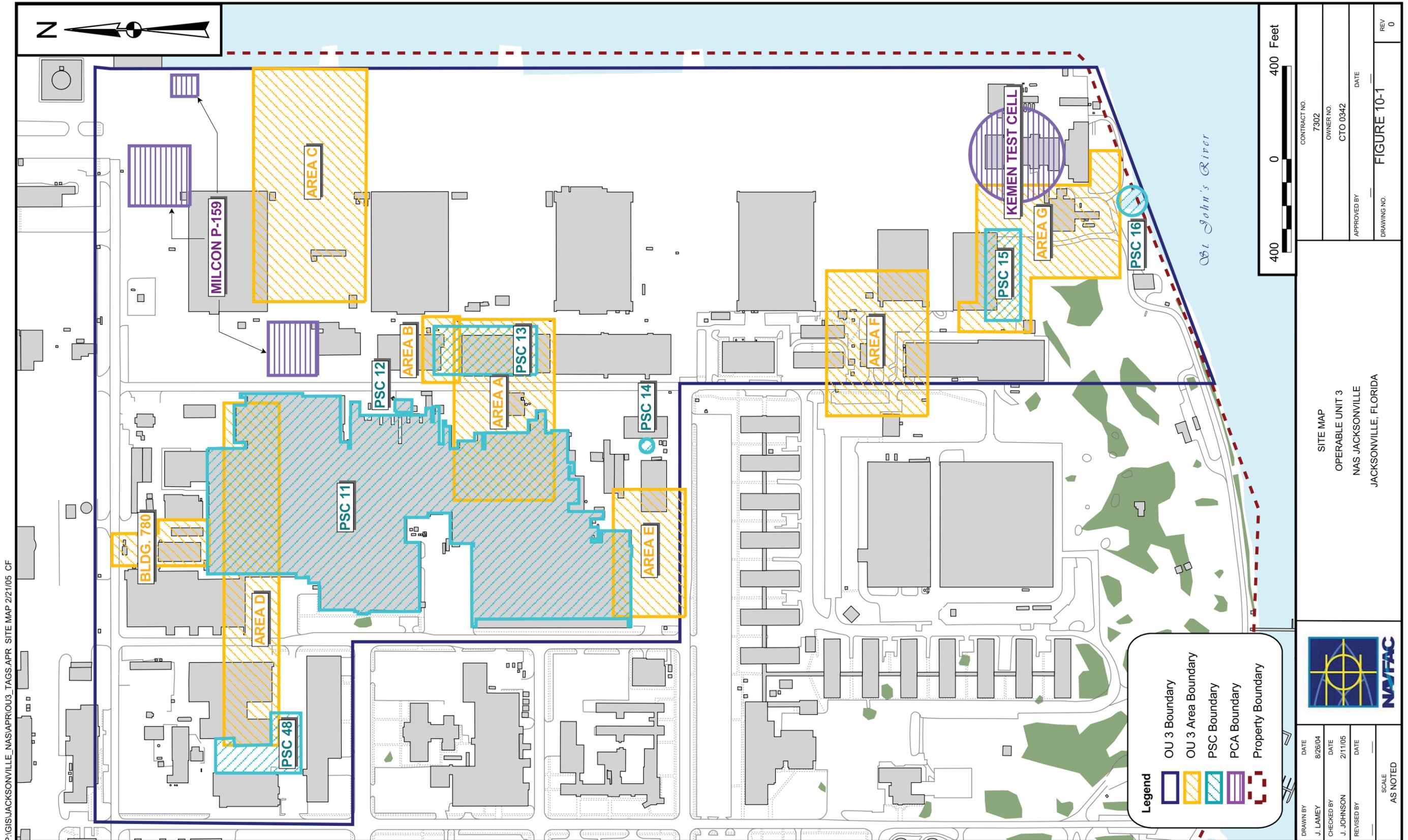
Aquatic receptors in the St. Johns River may have the greatest potential of a completed exposure pathway based on groundwater discharging to surface water directly from the aquifer or via storm sewer outfalls. The Florida Marine SWCTLs are conservatively based on both human health and ecological receptors and will be used as default criteria to evaluate potential unacceptable risk to aquatic receptors.

Because of limited pore water data in river sediments, uncertainty exists as to whether aquatic receptors in the St. Johns River may be exposed to unacceptable levels of groundwater contamination from the groundwater to surface water pathway and the groundwater to surface water via storm sewer pathway. As stated in Sections 10.5.3.1 and 10.5.3.2, additional pore water and surface water sampling will be performed to address this uncertainty. In addition, the establishment of alternate concentration limits (ACLs) will be evaluated in accordance with USEPA's policy on "*Use of Alternate Concentration Limits (ACLs) in Superfund Cleanups*," (OSWER 9200.4-39, July 2005). This policy allows for establishment of ACLs if "On the basis of measurements or projections, there is or will be no statistically significant increase of such constituents from such groundwater in such surface water at the point of entry or at any point where there is reason to believe accumulation of constituents may occur downstream". In addition to pore water and surface water VOC measurements, contaminant flux measurements will be made in areas where groundwater contamination is determined to be discharging from sediment pore water or sewer outfalls. The flux results will be evaluated in conjunction with the NAS Jacksonville groundwater

flow and transport model developed by USGS and related surface water flow models for the St. Johns River.

#### **10.5.5 Current and Potential Future Site Uses**

OU 3 is currently used for industrial purposes and mostly covered with buildings and concrete or asphalt pavement. NAS Jacksonville is not proposed for Base Realignment and Closure. Therefore, it is reasonable to assume that NAS Jacksonville and OU 3 will continue to be used for industrial or non-residential purposes for the foreseeable future and that the existing concrete and asphalt pavement will be maintained with land use controls to restrict any direct exposure pathway. Similarly, the groundwater at OU 3 is not used as a drinking water source and is not expected to be in the future.



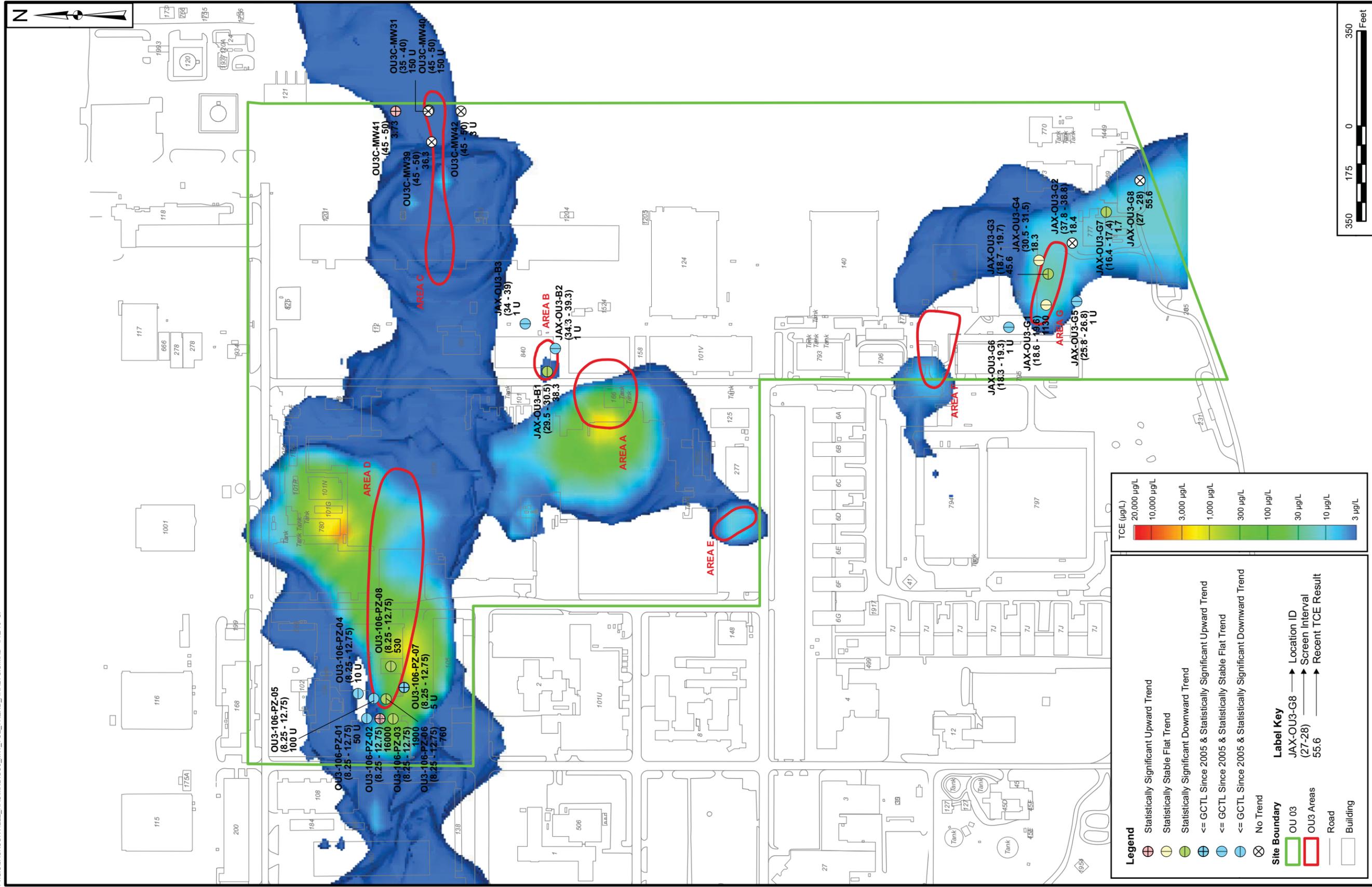
P:\GIS\JACKSONVILLE\_NAS\PROJ03\_TAGS\APR SITE MAP 2/21/05 CF



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REVISED BY		DATE	
SCALE	AS NOTED		

Source: TINUS, 2005. Five Year Review.

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**Legend**

- Statistically Significant Upward Trend
- Statistically Stable Flat Trend
- Statistically Significant Downward Trend
- <= GCTL Since 2005 & Statistically Significant Upward Trend
- <= GCTL Since 2005 & Statistically Stable Flat Trend
- <= GCTL Since 2005 & Statistically Significant Downward Trend
- No Trend

**Site Boundary**

- OU 03
- OU 3 Areas
- Road
- Building

**Label Key**

- JAX-OU3-G8 → Location ID
- (27-28) → Screen Interval
- 55.6 → Recent TCE Result

**TCE (µg/L)**

- 20,000 µg/L
- 10,000 µg/L
- 3,000 µg/L
- 1,000 µg/L
- 300 µg/L
- 100 µg/L
- 30 µg/L
- 10 µg/L
- 3 µg/L

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REVISED BY S. PAXTON		DATE 4/12/10	APPROVED BY	
SCALE AS NOTED		FIGURE NO. FIGURE 10-2		
LOCATION MAP AND TREND ANALYSIS RESULTS FOR TRICHLOROETHYLENE PLUME OU 3 NAS JACKSONVILLE JACKSONVILLE, FLORIDA				
		REV 0		



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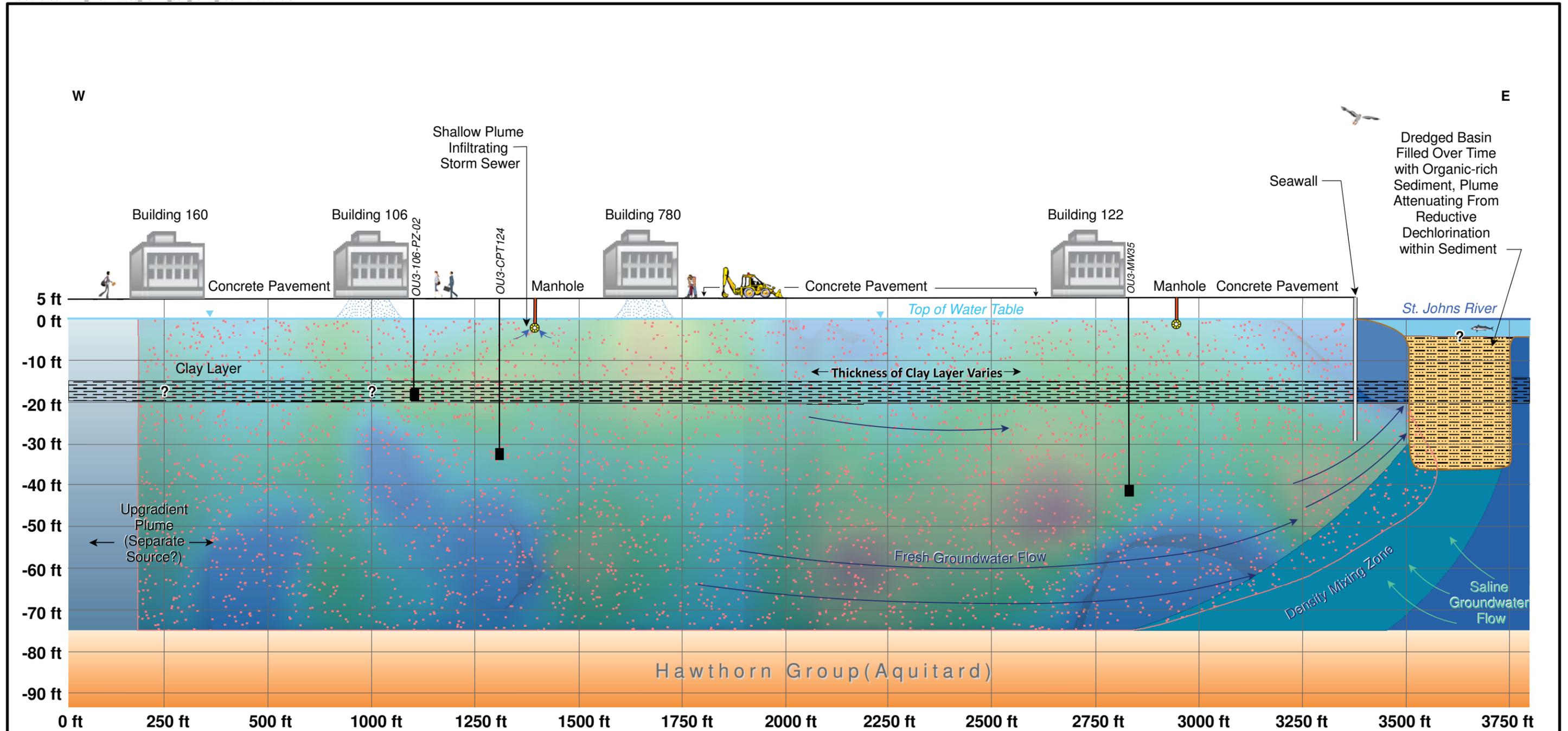
CROSS-SECTION PLAN VIEW  
 OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA



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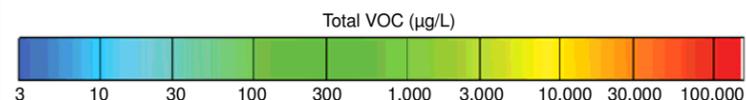
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**Legend**

- Storm Sewer
- Clay layer
- Plume



Note: This Conceptual Site Model is based on Total VOC data.

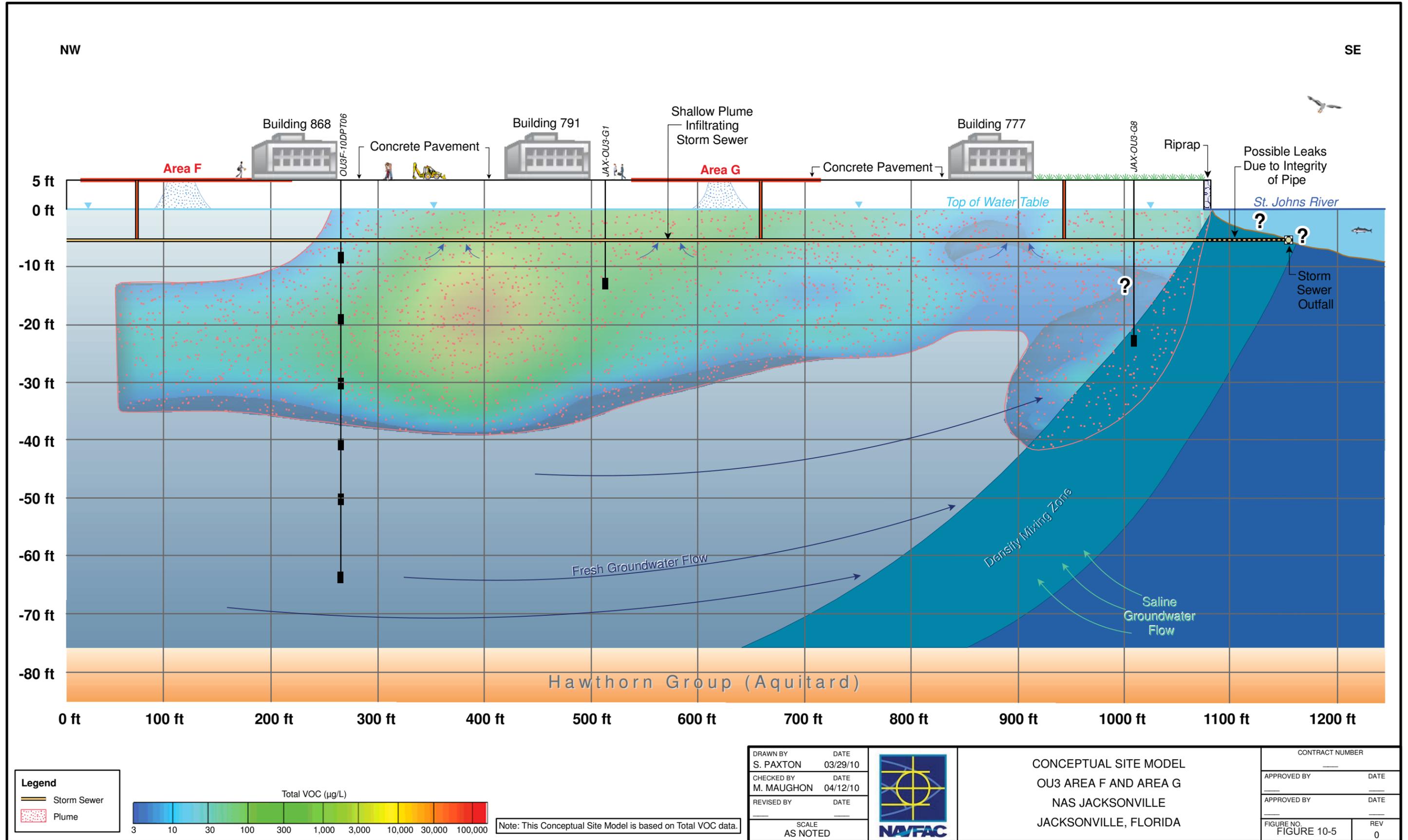
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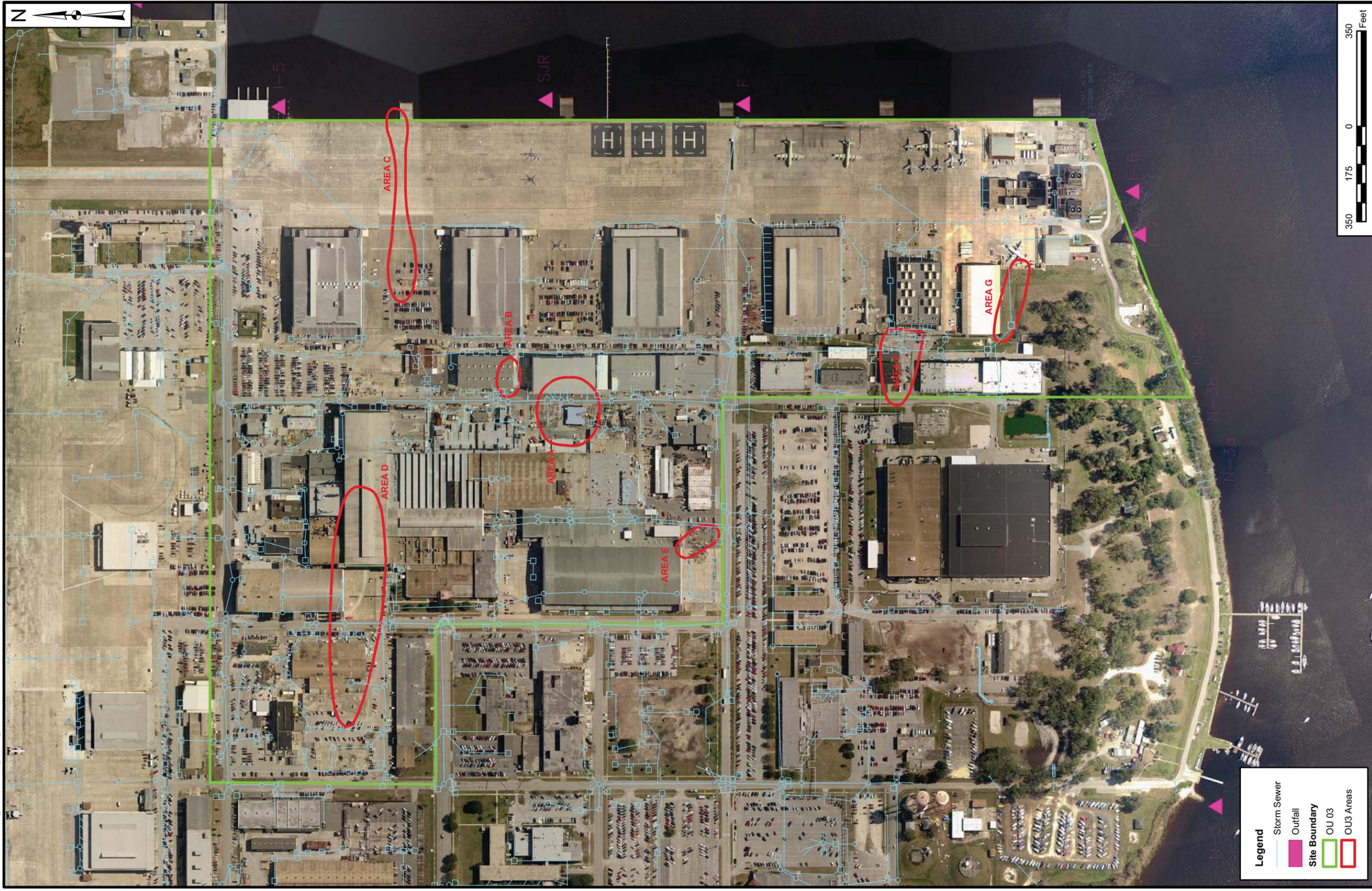


CONCEPTUAL SITE MODEL  
 OU3 BUILDINGS 106 AND 780 PLUME  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

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FIGURE NO. FIGURE 10-4	REV 0

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**Legend**

- Storm Sewer
- ▲ Outfall
- Site Boundary**
- OU 03
- OU3 Areas

CONTRACT NUMBER		REV	0
APPROVED BY	DATE	FIGURE NO.	FIGURE 10-6
APPROVED BY	DATE	REV	0

OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

OU3 STORM SEWER OUTFALL LOCATIONS



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S. PAXTON	4/07/09		
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S. PAXTON	4/06/10		

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132 ft<sup>3</sup>/day  
 0.00153 ft<sup>3</sup>/sec  
 0.69 gal/min

30 ft<sup>3</sup>/day  
 0.00035 ft<sup>3</sup>/sec  
 0.16 gal/min

200 ft<sup>3</sup>/day  
 0.00231 ft<sup>3</sup>/sec  
 1.04 gal/min



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REVISED BY	DATE
SCALE AS NOTED	



AREA G STORM SEWER INFILTRATION  
 BASED ON USGS GROUNDWATER MODELING  
 OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

CONTRACT NUMBER	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. FIGURE 10-7	REV 0

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

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

**11.1 PROBLEM DEFINITION**

Additional data is required to refine the CSM and prepare an RI/FS Addendum for OU 3. Additional data must be collected to determine if soil contamination is present at the boundary of OU 3, to determine the nature and extent of source area groundwater contamination upgradient of Building 106, and if contaminated groundwater is potentially discharging to the St. Johns River directly from the surficial aquifer or storm sewers. This data will be used to develop remedial alternatives for the FS, which will likely include an evaluation of LUCs, with a monitoring program for MNA, as well as active remedies, such that the NAS Jacksonville Partnering Team can select a final remedy for OU 3.

**11.2 INFORMATION INPUTS**

This sampling effort will utilize a Triad Approach to collect, evaluate, and prioritize data collection to evaluate the extent of contaminants in the subsurface. A MIP will be used to build on previously collected MIP data coverage and to provide information for real time decision making for the collection of groundwater samples by DPT for analysis of VOCs in a National Environmental Laboratory Accreditation Program (NELAP) approved mobile laboratory. In addition, surface soil sampling will be conducted to establish the boundaries of OU 3. Storm water data will also be collected from storm sewers near the outfalls at OU 3. Storm water and surface water data will be collected to evaluate tidal cycle effects on any groundwater target analytes that may be discharging from the southern storm sewer associated with Area G. Finally, sediment pore water and surface water samples will be collected from offshore areas where contaminated groundwater is thought to possibly be discharging into the St. Johns River.

The following physical and chemical data will be collected during this investigation:

1. Membrane Interface Probe: MIP is a screening tool with semi-quantitative capabilities acting as an interface between contaminants in the subsurface and gas phase detectors at the surface. MIP acquisition software logs detector signal with depth. The detectors to be utilized include an electron capture detector (ECD) and a flame ionization detector (FID). The ECD is designed for sensitivity to CVOCs and the FID is designed for sensitivity to straight chain hydrocarbons. The ECD and FID will be used for VOCs analysis.

2. Chemical Data: Groundwater, water in storm sewers, sediment pore water, surface water, and surface soil samples will be analyzed for the select list of target analytes that are presented in Worksheet #15. The sampling methods that will be utilized are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.
3. Field Parameters: Field investigation parameters for groundwater will include dissolved oxygen (DO), 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.
4. Groundwater Level Measurements: Synoptic groundwater levels will be measured in each monitoring well to determine the groundwater flow direction. The sampling methods are presented in Worksheet #18.
5. MNA Parameters: Groundwater will be analyzed for select MNA parameters, including iron and manganese (total and dissolved), dissolved gases (methane, ethane, ethene, and hydrogen), total organic carbon (TOC), anions (chloride, nitrate, nitrite, and sulfate), dissolved sulfide, alkalinity, and the presence of microbial populations that are known to assist in natural degradation processes. The sampling methods are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.
6. Project Action Limits (PALs): Concentrations of target analytes will be compared against PALs. The PALs for this RI Addendum are derived from the following criteria for each media of concern:

### **Surface Soil**

- Soil Cleanup Target Levels (SCTLs) for Florida Chapter 62-777, Florida Administrative Code (F.A.C.), Table II (Soils) - Residential only, not leachability.
- Derived Alternative Cleanup Target Levels (CTLs), Chapter 62-780 (5), F.A.C.
- Apportioned SCTLs Chapter 62-780 (2)(b)1.2.(V), F.A.C.
- 95% Upper Confidence Limit (UCL) Approach, Chapter 62-780.680(2)(b)1.e.(II), F.A.C.
- The laboratory practical quantitation limit (PQL) should be used if it is less stringent than the CTL according to Chapter 62-780.680(2)(b)2.a.(III), F.A.C. The PQL is the lowest concentration that a laboratory can accurately report on a chemical.

- NAS Jacksonville Basewide Background Concentrations (for metals).
- USEPA Regions 3, 6, and 9 (December, 2009 or more recent) Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites - Residential/Industrial Soil Values.

### **Groundwater**

- FDEP GCTLs, Chapter 62-777, F.A.C. Table I (Groundwater).
- Florida Drinking Water Standards, Chapter 62-550.310, F.A.C.
- The laboratory PQL should be used if it is less stringent than the CTL, according to Chapter 62-780.680(1)(c), F.A.C. The PQL is the lowest concentration that a laboratory can accurately report on a chemical.
- USEPA Maximum Concentration Levels (MCLs).
- NAS Jacksonville Basewide Background Concentrations (for metals).
- USEPA Regions 3, 6, and 9 (December, 2009 or more recent date) RSLs for Chemical Contaminants at Superfund Sites - Groundwater values.
- Chapter 62-780 F.A.C., Definition for low yield, poor quality aquifers.

### **Surface/Pore/Storm Water**

- FDEP Marine Surface Water Quality Classifications, Chapter 62-302.53 F.A.C.
- Region IV = USEPA Region IV Ecological Risk Assessment Bulletin - Supplement to RAGS. August 11, 1999. Saltwater Screening Values for Hazardous Waste Sites. Website updated 30 November 2001.
- Chapter 62-777 F.A.C., Marine SWCTL based on protection of aquatic life.
- Chapter 62-302.800 F.A.C., Site Specific Alternative Criteria (Note\*) For groundwater discharging to surface water that would allow calculation of an ACL.
- OSWER 9200.4-39, Use of ACLs in Superfund Cleanups.
- NAS Jacksonville National Pollutant Discharge Elimination System (NPDES) Permit.
- USEPA Regions 3, 6, and 9 (December, 2009 or more recent date) RSLs for Chemical Contaminants at Superfund Sites - Groundwater values.

### **11.3 STUDY AREA BOUNDARIES**

The media of interest for this SAP include surface soil, groundwater, sediment pore water, surface water, and storm sewer water. The study area boundaries are defined by the operational boundaries of OU 3 and the adjacent areas of the St. Johns River which could be impacted by the migration of contaminated groundwater directly to the river or into storm drains at OU 3 which have outfalls in the St. Johns River. The temporal boundaries that apply to each media are the time frames necessary to meet the Remedial Action Objectives that will be specified in the ROD for this site to be developed at a later date. The scale of the decision making is in general regards to site workers, potential future residents, and ecological receptors in the St. Johns River.

Data will be collected from the following populations of interest, and will be used for decision making during this investigation. The surface soil population of interest includes contaminated surface soil at the 0 to 2 feet bgs interval within the OU3 boundary. The groundwater population of interest includes contaminated groundwater within the surficial aquifer (less than 100 feet bgs) upgradient of Building 106 and that has migrated along flow paths and within the storm sewers and is potentially discharging to the St. Johns River. The sediment pore water population of interest includes pore water that is potentially impacted by contaminated groundwater that has migrated to the river. The surface water population of interest includes surface water within areas where contaminated groundwater discharges directly or through storm sewers to surface water (i.e., offshore at Areas C and G). The Trident Probe will be used to collect both sediment pore water and surface water data in these areas. The storm sewer water population of interest includes water within the eastern storm sewer in Area G and at all other storm sewer outfalls within OU 3 potentially discharging contaminants to the St. Johns River (Figure 10-6).

### **11.4 ANALYTIC APPROACH**

Separate decision rules have been created for each of the environmental media and migration pathways being investigated. The decision rules presented below may recommend MNA or related monitoring program. If MNA or a monitoring program is recommended, it will be evaluated in the FS and presented in another UFP-SAP.

#### **11.4.1 Surface Soil Decision Rule**

Individual surface soil concentrations will be compared to surface soil PALs. The surface soil decision flow chart is presented on Figure 11-1.

- If any surface soil target analyte concentration exceeds the PAL for that target analyte, additional data will be collected via step out samples 60 days after receipt of first phase analytical results.

Step out samples will be collected until the individual surface soil target analyte concentration from the step out sample no longer exceeds the PAL.

- If any surface soil target analyte concentration is less than the PAL for that analyte (or group of analytes), then the soil contamination has been delineated for that analyte (or group of analytes) and the application of LUCs will be presented and evaluated in the FS.

#### **11.4.2 Impacts from Groundwater to Surface Water Decision Rule**

Groundwater contaminants have been measured at concentrations exceeding surface water PALs in monitoring wells upgradient of direct groundwater recharge areas in the St. Johns River. Additional groundwater data must be collected from the area of plume discharge in order to determine if impacts from contaminated groundwater to surface water are occurring. Individual groundwater target analyte concentrations will be compared to the surface water PALs in order to select surface water contaminants of potential concern (COPCs) that will be used during the risk assessment and for decision making. Surface water and sediment pore water will be collected with the Trident Probe. The seepage rate of pore water discharging to surface water and pore water COC data will be collected at the sediment surface with the UltraSeep, if necessary, as described below. The decision flow chart is presented on Figure 11-2.

- If all groundwater target analyte concentrations at the closest onshore monitoring location upgradient of seep/discharge locations are less than the surface water PALs, then the NAS Jacksonville Partnering Team will develop an appropriate monitoring program to be included in the remedial alternatives that will be presented in the FS.
- If any groundwater target analyte concentration at the closest onshore monitoring location upgradient of the seep/discharge locations is greater than the surface water PAL, then pore water data (2 feet below the top of sediments) and surface water data (1 foot above the top of sediments) will be collected with the Trident Probe at the seep/discharge locations and compared to surface water PALs.
  - If the pore water target analyte concentrations do not exceed the PALs, then the NAS Jacksonville Partnering Team will establish ACLs and evaluate the most appropriate monitoring locations to be included in the remedial alternatives that will be presented in the FS.
  - If any pore water concentrations exceed the PALs, then pore water and seepage rate data will be collected at the sediment surface over a full tidal cycle using the UltraSeep.
    - If the pore water data at the sediment surface do not exceed the PALs, then the NAS Jacksonville Partnering Team will establish ACLs and evaluate the most

appropriate monitoring locations to be included in the remedial alternatives that will be presented in the FS.

- If the pore water data at the sediment surface exceed the PALs, then the NAS Jacksonville Partnering Team will calculate the mass flux of the groundwater COC discharge to surface water; perform a mixing analysis based on the COC mass flux, river flow, and groundwater COC concentrations measured in surface water; and predict the potential increase in target analyte concentrations in surface water within the area of groundwater discharge.
  - If the predicted increase in target analyte concentrations in surface water within the area of groundwater discharge is considered by the NAS Jacksonville Partnering Team to be negligible, then the NAS Jacksonville Partnering Team will establish ACLs and evaluate the most appropriate monitoring locations to be included in the remedial alternatives that will be presented in the FS.
  - If the predicted increase in target analyte concentrations in surface water within the area of groundwater discharge is not considered by the NAS Jacksonville Partnering Team to be negligible, then the NAS Jacksonville Partnering Team will conduct a risk assessment and evaluate monitoring and other groundwater remedial alternatives in the FS.

#### **11.4.3 Impacts from Groundwater to Eastern Sewer in Area G Decision Rule**

Storm sewer water has become contaminated as a result of contaminated groundwater infiltrating into the fractured storm sewer system. Storm water contaminants have been measured in upstream manholes of the eastern sewer in Area G at concentrations that exceed surface water PALs, particularly at low tide. Additional data must be collected from the storm sewer to determine if any impacts are occurring to surface water from contaminants in the storm water at the point of entry to surface water. Target analyte concentrations and sewer flow data will be collected from the closest sewer access point to the shoreline over a full tidal cycle and at the outfall using the Trident Probe as necessary at the submerged outfall. The decision flow chart is presented on Figure 11-3.

- If storm water target analyte concentrations of samples collected over the tidal cycle do not exceed the surface water PALs, then the NAS Jacksonville Partnering Team will develop an appropriate monitoring program to be included in the remedial alternatives that will be presented in the FS.
- If any storm water target analyte concentrations of samples collected over the tidal cycle exceeds the surface water PALs, then the NAS Jacksonville Partnering Team will calculate the mass flux

of the contaminant discharge to surface water; perform a mixing analysis based on the contaminant mass flux, river flow, and target analyte concentrations measured in surface water; and predict the potential increase in target analyte concentrations in surface water at the outfall area.

- If the predicted increase in concentrations in surface water at the outfall area is considered by the NAS Jacksonville Partnering Team to be negligible, then the NAS Jacksonville Partnering Team will establish ACLs and evaluate the most appropriate monitoring locations to be included in the remedial alternatives that will be presented in the FS.
- If the predicted increase in target analyte concentrations in surface water at the outfall area is not considered by the NAS Jacksonville Partnering Team to be negligible, then the NAS Jacksonville Partnering Team will conduct a risk assessment and evaluate monitoring and other groundwater remedial alternatives in the FS.

#### **11.4.4 Impacts from Groundwater to Sewer Decision Rule (Other Storm Sewers to be Surveyed)**

Similar to the eastern storm sewer in Area G, other storm sewers in OU 3 have the potential to intercept contaminated groundwater and discharge it to the St. Johns River. This may be the case for sewers that are leaking, pass through areas of shallow contaminated groundwater, and have invert elevations lower than the water table.

A survey will be completed to locate and sample any other storm sewers that discharge to the St. Johns River during dry periods (i.e., not during storm events). Storm sewers that discharge during dry periods are indicative of sewer systems that are leaking and receiving infiltration of groundwater. These sewers have the potential to discharge contaminants to the river if the infiltrated groundwater is contaminated. Sampling will occur only during low tide and will be conducted at the outfall or at the closest manhole or grated drain if the outfall is submerged or otherwise inaccessible. Samples will be analyzed in the on-site mobile laboratory for VOCs and compared to the surface water PALs. The decision flow chart is presented on Figure 11-4.

- If any storm sewer water target analyte concentrations exceed the surface water PAL, then additional storm sewer evaluation and sampling will be performed at a later date under a UFP-SAP Addendum that will be generated.
- If all storm sewer water target analyte concentrations are less than surface water PALs, then the Project Team will develop an appropriate monitoring program to be included in the remedial alternatives that will be presented in the FS.

#### **11.4.5 Monitored Natural Attenuation Decision Rule**

MNA parameters presented in Worksheet #17 will also be collected from sediment pore water, groundwater, and surface water to determine if conditions within these media are supporting, or will support, natural attenuation of contamination that is present in groundwater or that may migrate and reach the groundwater/surface water interface. This data will be used to determine if MNA is appropriate at particular locations within OU 3. The decision flow chart is presented on Figure 11-5.

- If the data indicate that natural attenuation is occurring or has the potential to occur, MNA will be presented as a component of the final remedy for OU 3.
- If the data indicate that natural attenuation will not be supported by current conditions, then MNA will not be presented as a component of the final remedy for OU 3.

**Figure 11-1 Surface Soil Decision Rule**

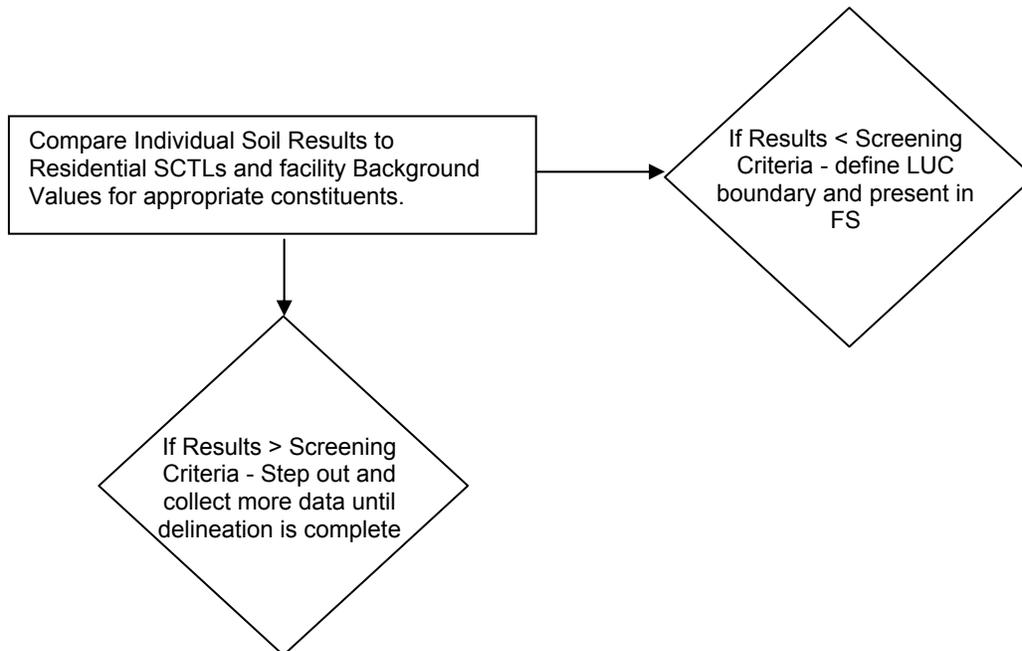


Figure 11-2 Groundwater to Surface Water Decision Rule

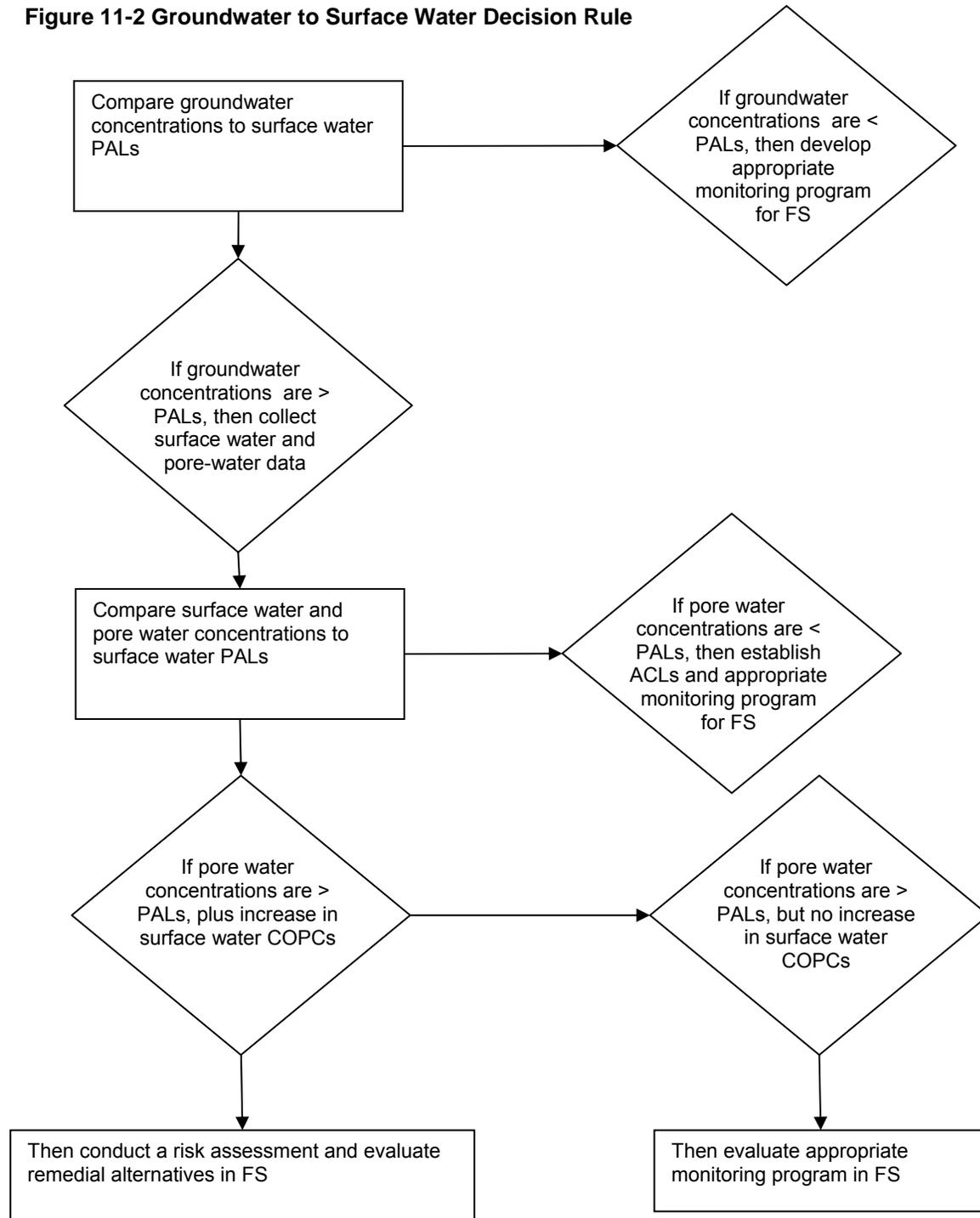


Figure 11-3 Groundwater Impacts to Eastern Sewer in Area G Decision Rule

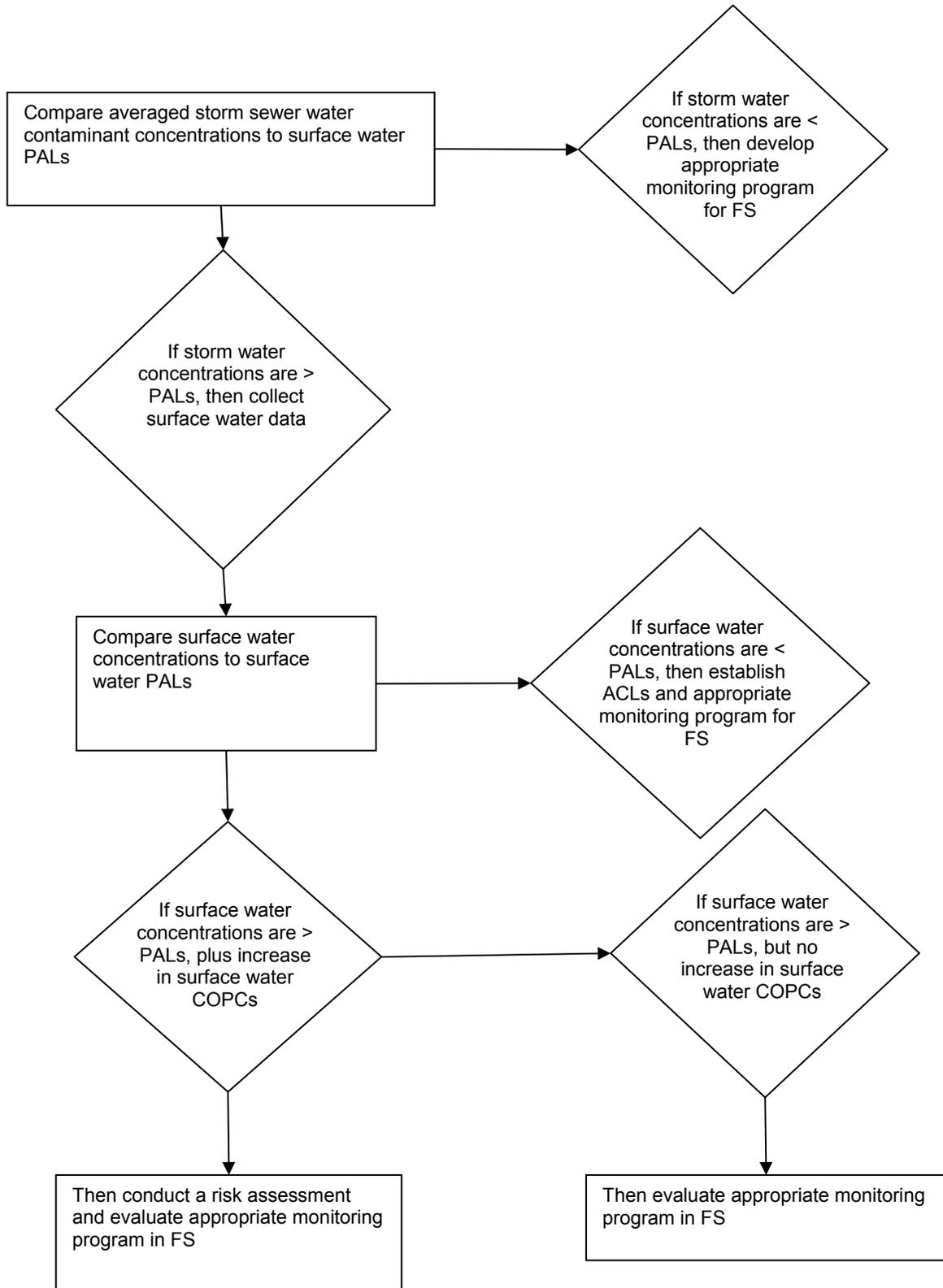


Figure 11-4 Groundwater to Sewer Decision Rule (Other Storm Sewers to be Surveyed)

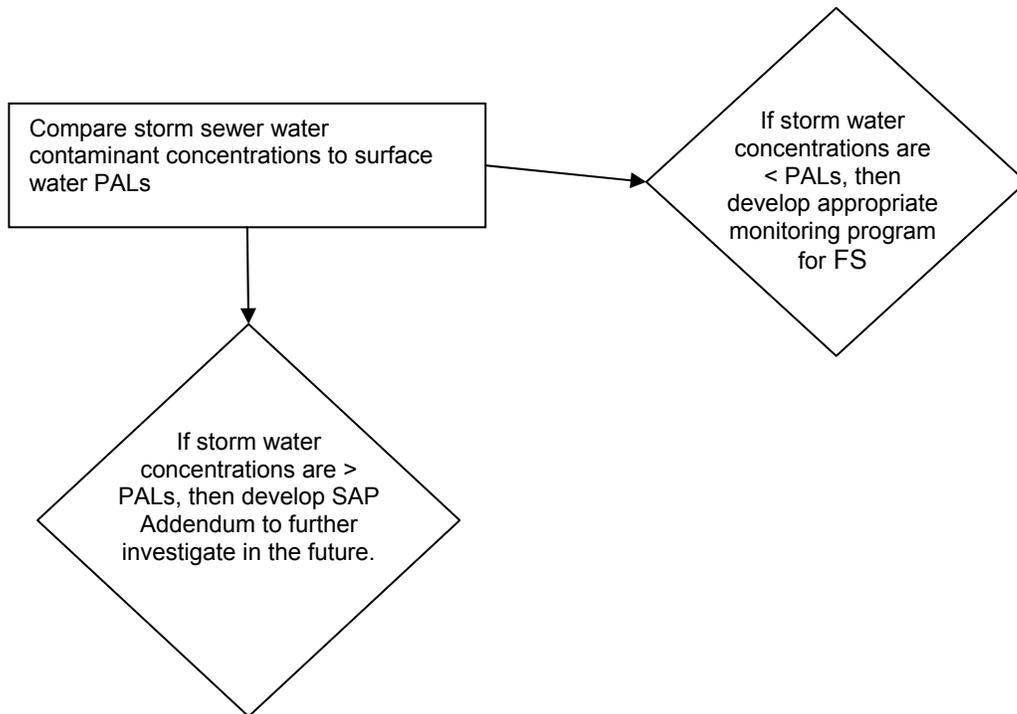
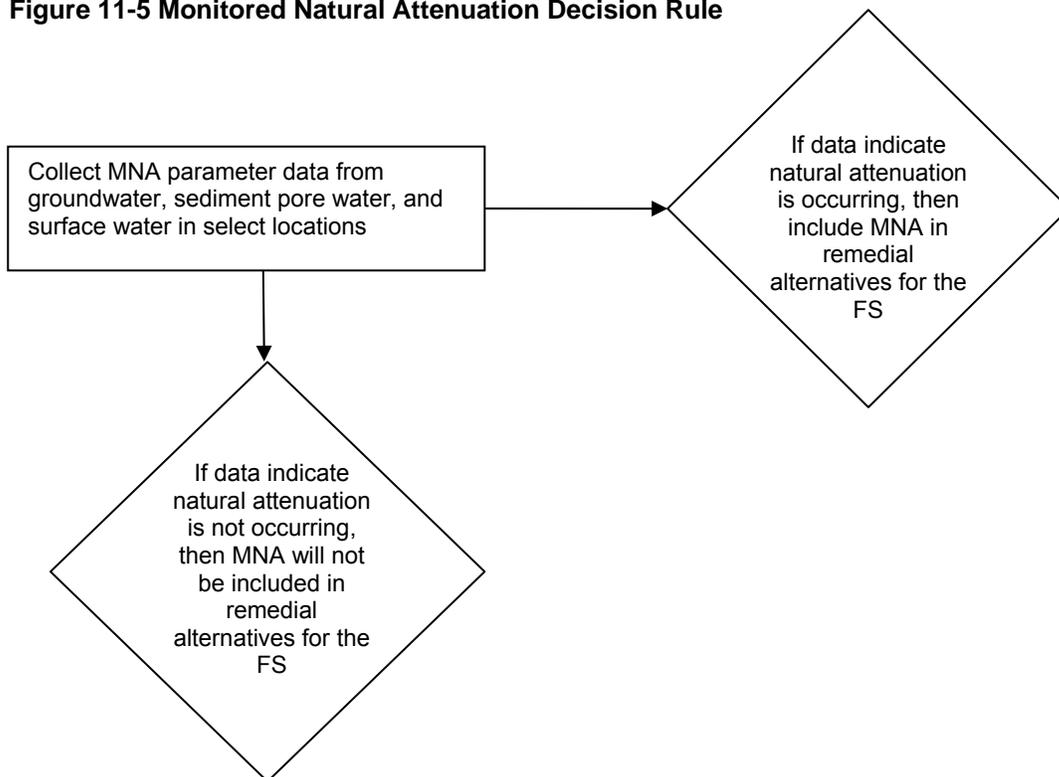


Figure 11-5 Monitored Natural Attenuation Decision Rule



## **11.5 MEASUREMENT AND PERFORMANCE CRITERIA**

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

## **11.6 PLAN FOR OBTAINING DATA**

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

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

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)
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment <sup>1</sup> .	Accuracy/ Bias/ Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	S & A
Trip Blanks	VOCs	One per cooler containing VOC samples.	Accuracy/ Bias/ Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	S & A
Field Duplicates	All Fractions	One per 10 field samples collected.	Precision	Values $>$ 5X LOQ: Relative Percent Difference (RPD) $\leq 30\%$ <sup>2, 3</sup> .	S & A
Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature between 2 and 6 degrees Celsius ( $4 \pm 2$ °C).	S

Notes:

- 1 – Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
- 2 – If duplicate values for non-metals are  $<$  5x LOQ, the absolute difference should be  $<$  2x LOQ.
- 3 – If duplicate values for metals are  $<$  5x LOQ, the absolute difference should be  $<$  4x LOQ.
- 4 – Miscellaneous parameters are being collected for monitoring natural attenuation and do not require any field QC samples except for a cooler blank.

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

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
None	Remedial Investigation/Feasibility Study for Operable Unit 3, April 2000	Harding Lawson Associates, groundwater, surface water, sediment, storm sewer/1993-1998	Decision Making: Historical data has been incorporated into the 3D computerized model that has been relied on for decision making in determining sample locations for this SAP. Since this SAP is for an RIFS Addendum, it also relies on historical data in the prior RIFS. Data from the prior RIFS may be utilized in the decision making process	The analytical data are not recent and will be supplemented with current information.

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

**14.1 FIELD INVESTIGATION TASK PLAN**

The field tasks are summarized below. A short description of these tasks is also provided.

- Mobilization/Demobilization
- Utility Clearance/Dig Permits
- Monitoring Equipment Calibration
- Membrane Interface Probe Sampling
- Surface Soil Sampling
- DPT Groundwater Sampling
- Pore Water/Surface Water Sampling
- Storm Sewer Sampling
- Well Installations
- Groundwater Sampling
- Water Level Measurements
- Investigation-Derived Waste (IDW) Management/Weekly Inspections
- Field Decontamination Procedures
- Field Documentation Procedures

Additional project activities include the following tasks:

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

**Mobilization/Demobilization**

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. Tetra Tech will coordinate with the Facility to identify locations for the storage of equipment and supplies. Site-specific Health and Safety Training for all Tetra Tech subcontractors will be provided as part of the site mobilization.

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 IDW generated during the conduct of the investigation.

### **Utility Clearance/Dig Permit**

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate utility clearance with the Facility and Sunshine State One Call. The Facility and Utility Companies subscribed to Sunshine State One Call will identify and mark-out utilities that may be present within the proposed well installation areas. Subsurface utilities will also be cleared by the well installation subcontractor by notifying the Sunshine State One Call utility clearing service. See Tetra Tech SOP HS-1.0 (Appendix A) on conducting well installations for further information. The FOL will also obtain a dig permit from the Public Works Department at NAS Jacksonville.

### **Monitoring Equipment Calibration**

These procedures are described in Worksheet #22.

### **Membrane Interface Probe Sampling**

A MIP probe will be used to collect field measurements of VOCs in the subsurface. The readings of VOCs will be accomplished by advancing a MIP detector into the subsurface via DPT methods continuously to target depths. A heating block and the tip of the probe will volatilize VOCs in the subsurface, which will defuse across a semi-permeable membrane to be withdrawn via a carrier gas to the surface where continuous measurements will be made via an ECD and a FID. Readings will be logged and a computer log generated for each sample location providing VOC distribution with depth in the subsurface.

### **Surface Soil Sampling**

Surface soil samples will be collected along the boundary of OU 3 (Figure 17-1) via hand auger in accordance with field SOPs provided in Appendix A. Soil samples obtained will be provided to the off-site laboratory Empirical Laboratory for VOCs, SVOCs, TRPH, and metals analysis. A second field event will be conducted for step out locations if warranted based on analytical results. The second phase field event will be conducted 60-days after receipt of analytical data. SOPs are detailed in Worksheets #18 and #21.

### **DPT Groundwater Sampling**

DPT methods will be used to collect in situ groundwater samples at select locations within OU 3. The

actual locations and depths will be determined after a review of MIP data. Samples will be analyzed in the mobile laboratory (KB Labs) for target CVOCs. This data will be used to verify data obtained from the MIP and to collect data in areas where MIP profiling is not possible due to subsurface refusal of the MIP probe.

DPT methods involve the advancement of a DPT sampling screen to a target depth. The screen is then revealed to the formation and groundwater is withdrawn via polyethylene tubing to the surface via a peristaltic pump. Samples will be collected via the straw method, placed into vials, and provided to the mobile laboratory (KB Labs) for analysis of target CVOCs.

### **Pore Water/Surface Water Sampling**

Pore water and surface water samples will be collected from areas in the St. Johns River suspected to receive discharge from contaminated groundwater using the Trident Probe system. The Trident Probe is a direct-push, integrated temperature sensor, conductivity sensor, and pore water sampler developed to screen sites for areas where groundwater may be discharging to a surface water body (Chadwick et al., 2003). Differences in observed conductivity and temperature indicate areas where groundwater discharge is occurring. After the area of groundwater discharge has been determined using the temperature and conductivity sensor probe, the integral pore water sampler probe can rapidly confirm the presence of freshwater or other chemical constituents. The Trident Probe sampler allows for advancement of a pore water sampling probe to a desired depth interval. The pore water sampler is typically used to collect pore water samples 2 feet below the sediment surface and surface water samples 1 foot above the sediment surface in locations where groundwater discharge is occurring.

Water enters the probe and then is pumped via polyethylene tubing to a peristaltic pump at the surface where samples are collected. Surface water samples are collected via the same procedures 1 foot above the sediment. The samples are collected within the probe via polyethylene tubing utilizing a peristaltic pump. The straw procedure will be utilized to collect samples to be placed into laboratory provided containers for analyses. Pore water analyses will be conducted by mobile laboratory (KB Labs) so that sample locations may be adjusted according to analytical results. Five percent of pore water samples will be replicated and analyzed by both the mobile laboratory (KB Labs) and the fixed-base laboratory (Empirical Laboratory). Samples for fixed-base laboratory analysis will be placed in a laboratory supplied sample cooler, chilled with ice, and shipped under chain-of-custody protocol to Empirical for analysis. Split samples will be evaluated to calculate a correlation between results.

In four to six representative locations (in the river offshore of both Area C and Area G) where target analytes in the pore water exceed one or more PALs (as determined from Trident Probe samples), the target analyte mass flux will be determined using the UltraSeep system to measure the seepage rate of

discharging groundwater and to collect a pore water sample at the surface of the sediment for VOC analysis (KB Labs) and microbial analyses (dehalococoides and reductase genes by Microbial Insights). The UltraSeep system is an integrated seepage meter and water sampling system for quantifying discharge rates and chemical loading from groundwater flow to coastal waters. Traditional seepage technology was modified and improved to include automated multiple sample collection and continuous flow detection with ultrasonic flow meters. The resultant instrument, the UltraSeep, makes direct measurements of advective flux and contaminant concentration at a particular location (Chadwick et al., 2003). The data produced are time series, over tidal cycles of groundwater flow contaminant concentration, and associated sensor data. These data allow an accurate determination of the presence or absence of groundwater flow and associated contaminant flux from a terrestrial site into a bay or estuary.

### **Storm Sewer Sampling**

Water samples will be collected from select storm sewer outfalls and via storm sewer manholes. Water samples will be collected via either Teflon sample buckets on an extension pole or via polyethylene tubing placed in the desired sampling location and then sampled via a peristaltic pump utilizing the straw method for placement of water samples into vials in accordance with field SOPs provided in Appendix A. For submerged outfalls, the Trident Probe will be used to collect pore water samples in the outfall area using the procedures previously described. Water samples obtained will be provided to the mobile laboratory (KB Labs) for VOC analysis. SOPs are detailed in Worksheets #18 and #21.

### **Well Installations**

Based on the results of MIP and DPT sampling, a second phase of field work will be conducted to install monitoring wells. The locations, number, and design of the wells will be determined after review of the MIP and DPT data. In general, the wells will be designed to confirm DPT/MIP results and to provide a sufficient monitoring well network for the evaluation of MNA. Drilling methods and procedures to install the wells will be standard industry practices in accordance with Navy requirements for monitoring well design and installation (NAVFAC SE, 1997). SOPs are detailed in Worksheets #18 and #21.

### **Groundwater Sampling**

Groundwater samples from monitoring wells will be collected using low-flow purging techniques (discharge rate of less than 1 liter per minute) with a peristaltic pump using Teflon™ tubing dedicated to each well. All monitoring well groundwater samples will be collected using the procedures specified in FS 2200, Groundwater Sampling (FDEP, 2008). Worksheets #17 and #18 specify the groundwater sample locations and target analytes for this investigation. Worksheet #23 specifies the analytical methods to be used.

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

The sample aliquots for semivolatile organic compounds (SVOCs), including measurements for low level polycyclic aromatic hydrocarbons (PAHs), and metals (and MNA Parameters, if they are included in the sampling event) analyses will be collected directly from the discharge side of the peristaltic pump following the quiescent sampling procedure. The sample aliquot for VOCs analysis will be collected last by slowly pulling the Teflon™ tubing out of the well to minimize agitation of the water in the monitoring well and then transferring the contents of the tubing to a VOC vial. After collection, the samples will be placed in a cooler, chilled with ice, and shipped under chain-of-custody protocol to Empirical for analysis. SOPs are detailed in Worksheets #18 and #21. To further support analysis for MNA, samples will be collected for microbial analysis from monitoring wells in representative upgradient and downgradient locations. Samples will be analyzed for dehalococoides and reductase genes by Microbial Insights; locations of wells shall be determined based on mobile laboratory (KB Labs) analytical results.

### **Water Level Measurements**

One synoptic round of electronic water-level measurements will be conducted at the site as part of each groundwater sampling event to provide information regarding groundwater flow patterns and hydraulic gradients. Water-level measurements will be completed within the shortest time possible on the same day, and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data sets. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or north side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement event and between each monitoring well. SOPs are detailed in Worksheets #18 and #21.

### **Investigation-Derived Waste Management**

Types of IDW generated during this investigation that could be potentially contaminated include excess soil and sediment material collected but not placed in the laboratory supplied sample jars, sampling equipment decontamination wastewaters, and personnel protective equipment (PPE) and clothing. Based on the historical site activities and types of contaminants present, none of these IDW materials is expected to present a significant risk to human health or the environment if properly managed. Excess soil and sediment will initially be placed in 55-gallon labeled, sealable steel drums provided by NAS

Jacksonville Public Works Department. The drums will be inspected weekly until picked up and transported by the Public Works Department to a secured area designated by the Navy. Proper disposal of these wastes will be performed by the Navy (or its designee) after the analytical results of the soil samples are received from the laboratory and reviewed. PPE and clothing will be wiped clean and disposed of in trash containers.

### **Field Decontamination Procedure**

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

### **Field Documentation Procedures**

Pre-preserved, certified-clean bottle ware will be supplied by the laboratories. 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 FS 1000, General Sampling Procedures (FDEP, 2008).

Field documentation will be performed in accordance with Tetra Tech SOP SA-6.3 (Appendix A). A summary of all field activities will be properly recorded 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 date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- 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 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.

### **Analytical Tasks**

Chemical analysis will be performed by a mobile laboratory (KB Labs) and a fixed-base laboratory (Empirical and Microbial Insights).

KB Labs is a current NELAP approved laboratory with the state of Florida. A copy of the laboratory certification for KB Labs can be found in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. KB Labs is expected to meet the PALs for VOCs to the extent identified in Worksheet #15. KB Labs will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheets #19 and #30. A minimum of 5 percent of the samples screened for VOCs by the on-site laboratory will be split and analyzed off-site by Empirical for definitive VOCs analysis. Mobile laboratory results are reported on a wet weight basis.

Empirical is a current Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) accredited laboratory. A copy of the laboratory certification for Empirical can be found in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. Empirical is expected to meet the PALs to the extent identified in Worksheet #15. Empirical will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheets #19 and #30. Copies of the Laboratory SOPs are included in Appendix B. All 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 files. This information will also be captured in the project database which will eventually be uploaded to NIRIS. Percent moisture information will also be captured in the Site Investigation report.

Microbial Insights will perform analysis for dehalococoides and reductase genes. DoD ELAP accreditation is not required for these analyses.

## **Data Management**

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

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

- **Data Tracking:** Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The 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.

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**

An RI Addendum will be prepared summarizing the results of all field activities and presenting all information collected. The RI Addendum will be provided to the NAS Jacksonville Partnering Team for review. After incorporation of NAS Jacksonville Partnering Team comments, a final RI Addendum will be prepared and submitted as part of a combined RI/FS Addendum Report.

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

**15.1 Matrix: Groundwater at All of OU3 and PCA 25**

**15.1.1 Analytical Group: VOCs**

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,1,1-TRICHLOROETHANE	71-55-6	200	FDEP Residential GCTL	67	1	0.5	0.25
<b>1,1,2,2-TETRACHLOROETHANE</b>	<b>79-34-5</b>	<b>0.2</b>	<b>FDEP Residential GCTL</b>	<b>0.067</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,1,2-TRICHLOROETHANE	79-00-5	5	FDEP Residential GCTL	1.7	1	0.5	0.25
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	210,000	FDEP Residential GCTL	70,000	1	0.5	0.25
1,1-DICHLOROETHANE	75-34-3	70	FDEP Residential GCTL	23	1	0.5	0.25
1,1-DICHLOROETHENE	75-35-4	7	FDEP Residential GCTL	2.3	1	0.5	0.25
1,2,4-TRICHLOROBENZENE	120-82-1	70	FDEP Residential GCTL	23	1	0.5	0.25
<b>1,2-DIBROMO-3-CHLOROPROPANE</b>	<b>96-12-8</b>	<b>0.2</b>	<b>FDEP Residential GCTL</b>	<b>0.067</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
<b>1,2-DIBROMOETHANE</b>	<b>106-93-4</b>	<b>0.02</b>	<b>FDEP Residential GCTL</b>	<b>0.0067</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
1,2-DICHLOROBENZENE	95-50-1	600	FDEP Residential GCTL	200	1	0.5	0.25
1,2-DICHLOROETHANE	107-06-2	3	FDEP Residential GCTL	1.0	1	0.5	0.25
1,2-DICHLOROPROPANE	78-87-5	5	FDEP Residential GCTL	1.7	1	0.5	0.25
1,3-DICHLOROBENZENE	541-73-1	210	FDEP Residential GCTL	70	1	0.5	0.25
1,4-DICHLOROBENZENE	106-46-7	75	FDEP Residential GCTL	25	1	0.5	0.25
2-BUTANONE	78-93-3	4,200	FDEP Residential GCTL	1,400	10	5	2.5
2-HEXANONE	591-78-6	280	FDEP Residential GCTL	93	10	5	2.5
4-METHYL-2-PENTANONE	108-10-1	560	FDEP Residential GCTL	190	10	5	2.5
ACETONE	67-64-1	6,300	FDEP Residential GCTL	2,100	10	5	2.5
BENZENE	71-43-2	1	FDEP Residential GCTL	0.33	1	0.5	0.25
<b>BROMODICHLOROMETHANE</b>	<b>75-27-4</b>	<b>0.6</b>	<b>FDEP Residential GCTL</b>	<b>0.20</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
BROMOFORM	75-25-2	4.4	FDEP Residential GCTL	1.5	1	0.5	0.25

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
BROMOMETHANE	74-83-9	9.8	FDEP Residential GCTL	3.3	1	0.5	0.25
CARBON DISULFIDE	75-15-0	700	FDEP Residential GCTL	230	1	0.5	0.25
CARBON TETRACHLORIDE	56-23-5	3	FDEP Residential GCTL	1.0	1	0.5	0.25
CHLOROETHANE	108-90-7	100	FDEP Residential GCTL	33	1	0.5	0.25
<b>CHLORODIBROMOMETHANE</b>	<b>124-48-1</b>	<b>0.4</b>	<b>FDEP Residential GCTL</b>	<b>0.13</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
CHLOROETHANE	75-00-3	12	FDEP Residential GCTL	4.0	1	0.5	0.25
CHLOROFORM	67-66-3	70	FDEP Residential GCTL	23	1	0.5	0.25
CHLOROMETHANE	74-87-3	2.7	FDEP Residential GCTL	0.90	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP Residential GCTL	23	1	0.5	0.25
CIS-1,3-DICHLOROPROPENE	10061-01-5	NA	---	NA	1	0.5	0.25
CYCLOHEXANE	110-82-7	NA	---	NA	1	0.5	0.25
DICHLORODIFLUOROMETHANE	75-71-8	1,400	FDEP Residential GCTL	470	1	0.5	0.25
ETHYLBENZENE	100-41-4	30	FDEP Residential GCTL	10	1	0.5	0.25
<b>ISOPROPYLBENZENE</b>	<b>98-82-8</b>	<b>0.8</b>	<b>FDEP Residential GCTL</b>	<b>0.27</b>	<b>1</b>	<b>0.5</b>	<b>0.25</b>
TOTAL XYLENES	1330-20-7	20	FDEP Residential GCTL	6.7	5	2.5	1
METHYL ACETATE	79-20-9	3,000	FDEP Residential GCTL	1,000	5	2.5	1
METHYL TERT-BUTYL ETHER	1634-04-4	20	FDEP Residential GCTL	6.7	1	0.5	0.25
METHYLENE CHLORIDE	75-09-2	5	FDEP Residential GCTL	1.7	3	1.5	1
METHYL CYCLOHEXANE	108-87-2	NA	---	NA	1	0.5	0.25
STYRENE	100-42-5	100	FDEP Residential GCTL	33	1	0.5	0.25
TETRACHLOROETHENE	127-18-4	3	FDEP Residential GCTL	1.0	1	0.5	0.25
TOLUENE	108-88-3	40	FDEP Residential GCTL	13	1	0.5	0.25
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FDEP Residential GCTL	33	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NA	---	NA	1	0.5	0.25

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
TRICHLOROETHENE	79-01-6	3	FDEP Residential GCTL	1.0	1	0.5	0.25
TRICHLOROFLUOROMETHANE	75-69-4	2,100	FDEP Residential GCTL	700	5	2.5	1
VINYL CHLORIDE	75-01-4	1	FDEP Residential GCTL	0.33	1	0.5	0.25

**Mobile Laboratory**  
**Matrix: Groundwater**  
**Analytical Group: VOCs**

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	KB Labs		
					LOQ (µg/L)	LOD (µg/L)	MDL (µg/L)
<i>cis</i> -1,2-Dichloroethene	156-59-2	70	FDEP Residential GCTL	23	1.0	1.0	0.28
<i>trans</i> -1,2-Dichloroethene	156-60-5	100	FDEP Residential GCTL	33	1.0	1.0	0.20
Isopropyl benzene	98-82-8	0.8	FDEP Residential GCTL	0.27	0.5	0.5	0.19
Tetrachloroethene	127-18-4	3	FDEP Residential GCTL	1.0	1.0	1.0	0.18
Trichloroethene	79-01-6	3	FDEP Residential GCTL	1.0	1.0	1.0	0.26
1,1,1-Trichloroethane	71-55-6	200	FDEP Residential GCTL	67	1.0	1.0	0.49
1,1-Dichloroethane	75-34-3	70	FDEP Residential GCTL	23	1.0	1.0	0.40
1,1-Dichloroethene	75-35-4	7	FDEP Residential GCTL	2.3	1.0	1.0	0.45
1,2-Dichloroethane	107-06-2	3	FDEP Residential GCTL	1.0	1.0	1.0	0.20
Chloroethane	75-00-3	12	FDEP Residential GCTL	4.0	1.0	1.0	0.37
Toluene	108-88-3	40	FDEP Residential GCTL	13	1.0	1.0	0.17
Vinyl Chloride	75-01-4	1	FDEP Residential GCTL	0.33	1.0	1.0	0.21

**Matrix: Groundwater at Building 101S in OU3**  
**15.1.2 Analytical Group: SVOCs and Low Level PAHs**

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,1-BIPHENYL	92-52-4	0.5	FDEP Residential GCTL	0.17	5	2.5	0.5
2,4,5-TRICHLOROPHENOL	95-95-4	1	FDEP Residential GCTL	0.33	5	2.5	1
2,4,6-TRICHLOROPHENOL	88-06-2	3.2	FDEP Residential GCTL	1.1	5	2.5	1
2,4-DICHLOROPHENOL	120-83-2	0.3	FDEP Residential GCTL	0.10	5	2.5	1
2,4-DIMETHYLPHENOL	105-67-9	140	FDEP Residential GCTL	47	5	2.5	1
2,4-DINITROPHENOL	51-28-5	14	FDEP Residential GCTL	4.7	10	5	2.5
2,4-DINITROTOLUENE	121-14-2	0.05	FDEP Residential GCTL	0.017	5	2.5	1
2,6-DINITROTOLUENE	606-20-2	0.05	FDEP Residential GCTL	0.017	5	2.5	1
2-CHLORONAPHTHALENE	91-58-7	560	FDEP Residential GCTL	190	5	2.5	1
2-CHLOROPHENOL	95-57-8	35	FDEP Residential GCTL	12	5	2.5	1
2-METHYLNAPHTHALENE (1)	91-57-6	28	FDEP Residential GCTL	9.3	0.2	0.1	0.05
2-METHYLPHENOL	95-48-7	35	FDEP Residential GCTL	12	5	2.5	1
2-NITROANILINE	88-74-4	21	FDEP Residential GCTL	7.0	5	2.5	1
2-NITROPHENOL	88-75-5	56	FDEP Residential GCTL	19	5	2.5	1
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	0.5	FDEP Residential GCTL	0.17	5	2.5	1
3,3'-DICHLOROBENZIDINE	91-94-1	0.08	FDEP Residential GCTL	0.028	5	2.5	1
3-NITROANILINE	99-09-2	1.7	FDEP Residential GCTL	0.57	5	2.5	1
4,6-DINITRO-2-METHYLPHENOL	534-52-1	0.7	FDEP Residential GCTL	0.23	10	5	2.5
4-BROMOPHENYL PHENYL ETHER	101-55-3	70	FDEP Residential GCTL	23	5	2.5	1
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	2.1	FDEP Residential GCTL	0.70	5	2.5	1
4-CHLORO-3-METHYLPHENOL	59-50-7	63	FDEP Residential GCTL	21	5	2.5	1

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
4-CHLOROANILINE	106-47-8	28	FDEP Residential GCTL	9.3	5	2.5	1
<b>4-METHYLPHENOL</b>	<b>106-44-5</b>	<b>3.5</b>	<b>FDEP Residential GCTL</b>	<b>1.2</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>4-NITROANILINE</b>	<b>100-01-6</b>	<b>1.7</b>	<b>FDEP Residential GCTL</b>	<b>0.57</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
4-NITROPHENOL	100-02-7	56	FDEP Residential GCTL	19	5	2.5	1
ACENAPHTHENE	83-32-9	20	FDEP Residential GCTL	6.7	5	2.5	1
ACENAPHTHYLENE	208-96-8	210	FDEP Residential GCTL	70	5	2.5	1
ACETOPHENONE	98-86-2	700	FDEP Residential GCTL	230	5	2.5	1
ANTHRACENE	120-12-7	2,100	FDEP Residential GCTL	700	5	2.5	1
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>3</b>	<b>FDEP Residential GCTL</b>	<b>1.0</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
BENZALDEHYDE	100-52-7	700	FDEP Residential GCTL	230	5	2.5	1
<b>BENZO(A)ANTHRACENE (1)</b>	<b>56-55-3</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
BENZO(A)PYRENE (1)	50-32-8	0.2	FDEP Residential GCTL	0.067	0.2	0.1	0.05
<b>BENZO(B)FLUORANTHENE (1)</b>	<b>205-99-2</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
BENZO(G,H,I)PERYLENE (1)	191-24-2	210	FDEP Residential GCTL	70	0.2	0.1	0.05
BENZO(K)FLUORANTHENE (1)	207-08-9	0.5	FDEP Residential GCTL	0.17	0.2	0.1	0.05
BIS(2-CHLOROETHOXY)METHANE	111-91-1	21	FDEP Residential GCTL	7.0	5	2.5	1
<b>BIS(2-CHLOROETHYL)ETHER</b>	<b>111-44-4</b>	<b>0.03</b>	<b>FDEP Residential GCTL</b>	<b>0.010</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	6	FDEP Residential GCTL	2.0	5	2.5	1
BUTYL BENZYL PHTHALATE	85-68-7	140	FDEP Residential GCTL	47	5	2.5	1
CAPROLACTAM	105-60-2	NA	---	NA	5	2.5	1
<b>CARBAZOLE</b>	<b>86-74-8</b>	<b>1.8</b>	<b>FDEP Residential GCTL</b>	<b>0.60</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>CHRYSENE</b>	<b>218-01-9</b>	<b>4.8</b>	<b>FDEP Residential GCTL</b>	<b>1.6</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>DIBENZO(A,H)ANTHRACENE (1)</b>	<b>53-70-3</b>	<b>0.005</b>	<b>FDEP Residential GCTL</b>	<b>0.0017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
DIBENZOFURAN	132-64-9	28	FDEP Residential GCTL	9.3	5	2.5	1
DIETHYL PHTHALATE	84-66-2	5,600	FDEP Residential GCTL	1,900	5	2.5	1
DIMETHYL PHTHALATE	131-11-3	70,000	FDEP Residential GCTL	23,000	5	2.5	1
DI-N-BUTYL PHTHALATE	84-74-2	700	FDEP Residential GCTL	230	5	2.5	1
DI-N-OCTYL PHTHALATE	117-84-0	140	FDEP Residential GCTL	47	5	2.5	1
FLUORANTHENE	206-44-0	280	FDEP Residential GCTL	93	5	2.5	1
FLUORENE	86-73-7	280	FDEP Residential GCTL	93	5	2.5	1
<b>HEXACHLOROBENZENE</b>	<b>118-74-1</b>	<b>1</b>	<b>FDEP Residential GCTL</b>	<b>0.33</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>HEXACHLOROBUTADIENE</b>	<b>87-68-3</b>	<b>0.4</b>	<b>FDEP Residential GCTL</b>	<b>1.3</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
HEXACHLOROCYCLOPENTADIENE	77-47-4	50	FDEP Residential GCTL	17	5	2.5	1
<b>HEXACHLOROETHANE</b>	<b>67-72-1</b>	<b>2.5</b>	<b>FDEP Residential GCTL</b>	<b>0.83</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>INDENO(1,2,3-CD)PYRENE (1)</b>	<b>193-39-5</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
ISOPHORONE	78-59-1	37	FDEP Residential GCTL	12	5	2.5	1
NAPHTHALENE	91-20-3	14	FDEP Residential GCTL	4.7	5	2.5	1
<b>NITROBENZENE</b>	<b>98-95-3</b>	<b>3.5</b>	<b>FDEP Residential GCTL</b>	<b>1.2</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.005</b>	<b>FDEP Residential GCTL</b>	<b>0.0017</b>	<b>5</b>	<b>2.5</b>	<b>1</b>
N-NITROSODIPHENYLAMINE	86-30-6	7.1	FDEP Residential GCTL	2.7	5	2.5	1
<b>PENTACHLOROPHENOL</b>	<b>87-86-5</b>	<b>1</b>	<b>FDEP Residential GCTL</b>	<b>0.33</b>	<b>20</b>	<b>10</b>	<b>2</b>
PHENANTHRENE	85-01-8	210	FDEP Residential GCTL	70	5	2.5	1
PHENOL	108-95-2	10	FDEP Residential GCTL	3.3	5	2.5	1
PYRENE	129-00-0	210	FDEP Residential GCTL	70	5	2.5	1

Note:

(1) 8270D Low Level SOP utilized for PAHs.

Matrix: Groundwater at PCA 25

15.1.3 Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1-METHYLNAPHTHALENE	90-12-0	28	FDEP Residential GCTL	9.3	0.2	0.1	0.05
2-METHYLNAPHTHALENE	91-57-6	28	FDEP Residential GCTL	9.3	0.2	0.1	0.05
ACENAPHTHENE	83-32-9	20	FDEP Residential GCTL	6.7	0.2	0.1	0.05
ACENAPHTHYLENE	208-96-8	210	FDEP Residential GCTL	70	0.2	0.1	0.05
ANTHRACENE	120-12-7	2,100	FDEP Residential GCTL	700	0.2	0.1	0.05
<b>BENZO(A)ANTHRACENE</b>	<b>56-55-3</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
BENZO(A)PYRENE	50-32-8	0.2	FDEP Residential GCTL	0.067	0.2	0.1	0.05
<b>BENZO(B)FLUORANTHENE</b>	<b>205-99-2</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
BENZO(G,H,I)PERYLENE	191-24-2	210	FDEP Residential GCTL	70	0.2	0.1	0.05
BENZO(K)FLUORANTHENE	207-08-9	0.5	FDEP Residential GCTL	0.17	0.2	0.1	0.05
CHRYSENE	218-01-9	4.8	FDEP Residential GCTL	1.6	0.2	0.1	0.05
DIBENZO(A,H)ANTHRACENE	53-70-3	0.2	FDEP Residential GCTL	0.067	0.2	0.1	0.05
FLUORANTHENE	206-44-0	280	FDEP Residential GCTL	93	0.2	0.1	0.05
FLUORENE	86-73-7	280	FDEP Residential GCTL	93	0.2	0.1	0.05
<b>INDENO(1,2,3-CD)PYRENE</b>	<b>193-39-5</b>	<b>0.05</b>	<b>FDEP Residential GCTL</b>	<b>0.017</b>	<b>0.2</b>	<b>0.1</b>	<b>0.05</b>
NAPHTHALENE	91-20-3	14	FDEP Residential GCTL	4.7	0.2	0.1	0.05
PHENANTHRENE	85-01-8	210	FDEP Residential GCTL	70	0.2	0.1	0.05
PYRENE	129-00-0	210	FDEP Residential GCTL	70	0.2	0.1	0.05

**Matrix: Groundwater at PCA 25 and Building 101S in OU3**

**15.1.4 Analytical Group: Metals**

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
ALUMINUM	7429-90-5	200	FDEP Residential GCTL	67	200	100	50
ANTIMONY (2)	7440-36-0	6	FDEP Residential GCTL	2.0	4	2	1.3
ARSENIC	7440-38-2	10	FDEP Primary Standard	3.3	6	3	1.5
BARIUM	7440-39-3	2,000	FDEP Residential GCTL	670	40	20	10
BERYLLIUM (2)	7440-41-7	4	FDEP Residential GCTL	1.3	2	1	0.5
CADMIUM	7440-43-9	5	FDEP Residential GCTL	1.7	2	1	0.5
CALCIUM	7440-70-2	NA	---	NA	5,000	2,500	1,000
CHROMIUM	7440-47-3	100	FDEP Residential GCTL	33	10	5	2.5
COBALT	7440-48-4	140	FDEP Residential GCTL	47	15	10	5
COPPER	7440-50-8	1,000	FDEP Residential GCTL	330	10	5	2.5
IRON	7439-89-6	300	FDEP Residential GCTL	100	100	50	25
LEAD	7439-92-1	15	FDEP Residential GCTL	5.0	5	2.5	1.5
MAGNESIUM	7439-95-4	NA	---	NA	5,000	2,500	1,000
MANGANESE	7439-96-5	50	FDEP Residential GCTL	17	15	10	5
MERCURY	7439-97-6	2	FDEP Residential GCTL	0.67	0.4	0.2	0.1
MOLYBDENUM	7439-98-7	35	FDEP Residential GCTL	12	20	10	5
NICKEL	7440-02-0	100	FDEP Residential GCTL	33	10	5	2.5
POTASSIUM	9/7/7440	NA	---	NA	5,000	2,500	1,000
SELENIUM	7782-49-2	50	FDEP Residential GCTL	17	10	5	2.5
SILVER	7440-22-4	100	FDEP Residential GCTL	33	5	2.5	1

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
SODIUM	7440-23-5	160,000	FDEP Residential GCTL	53,000	5,000	2,500	1,000
THALLIUM (2)	7440-28-0	2	FDEP Primary Standard	0.67	2	1	0.7
VANADIUM	7440-62-2	49	FDEP Residential GCTL	16	15	10	5
ZINC	7440-66-6	5,000	FDEP Residential GCTL	1,700	20	10	5

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

**Matrix: Groundwater at PCA 25**  
**15.1.5 Analytical Group: Lead**

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
LEAD	7439-92-1	15	FDEP Residential GCTL	5.0	5	2.5	1.5

**15.2 Matrix: Surface Soil**  
**15.2.1 Analytical Group: Metals**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
ALUMINIUM	7429-90-5	80,000	FDEP Residential SCTL	27,000	40	20	10
ANTIMONY	7440-36-0	5.4	FDEP Leachability SCTL	1.8	2	1.6	1
ARSENIC	7440-38-2	2.1	FDEP Residential SCTL	0.70	2	1.2	0.6
BARIIUM	7440-39-3	120	FDEP Residential SCTL	40	8	2	1
BERYLLIUM	7440-41-7	63	FDEP Leachability SCTL	21	1	0.4	0.2
CADMIUM	7440-43-9	7.5	FDEP Leachability SCTL	2.5	1	0.4	0.2
CALCIUM	7440-70-2	NA	---	NA	1,000	400	200
CHROMIUM	7440-47-3	38	FDEP Leachability SCTL	13	2	0.8	0.4
COBALT	7440-48-4	1,700	FDEP Residential SCTL	570	2.5	2	1
COPPER	7440-50-8	150	FDEP Residential SCTL	50	2	1.6	0.8
IRON	7439-89-6	53,000	FDEP Residential SCTL	18,000	20	12	6
LEAD	7439-92-1	400	FDEP Residential SCTL	130	1	0.6	0.3

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
MAGNESIUM	7439-95-4	NA	---	NA	1,000	600	300
MANGANESE	7439-96-5	3,500	FDEP Residential SCTL	1,200	3	1.2	0.6
MERCURY	7439-97-6	2.1	FDEP Leachability SCTL	0.70	0.05	0.025	0.013
MOLYBDENUM	7439-98-7	440	FDEP Residential SCTL	150	1	0.5	0.25
NICKEL	7440-02-0	130	FDEP Leachability SCTL	110	2	1.2	0.6
POTASSIUM	9/7/7440	NA	---	NA	1,000	600	300
SELENIUM	7782-49-2	5.2	FDEP Leachability SCTL	1.7	2	1	0.5
SILVER	7440-22-4	17	FDEP Leachability SCTL	5.7	2	0.4	0.2
SODIUM	7440-23-5	NA	---	NA	1,000	600	300
THALLIUM	7440-28-0	2.8	FDEP Leachability SCTL	0.93	1.6	0.8	0.4
VANADIUM	7440-62-2	67	FDEP Residential SCTL	22	2.5	2	1
ZINC	7440-66-6	26,000	FDEP Residential SCTL	8,700	4	2	1

**Matrix: Surface Soil**

**15.2.2 Analytical Group: SVOCs and Low Level PAHs**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1,1-BIPHENYL	92-52-4	0.2	FDEP Leachability SCTL	0.067	0.333	0.133	0.030
2,4,5-TRICHLOROPHENOL	95-95-4	0.07	FDEP Leachability SCTL	0.023	0.333	0.133	0.030
2,4,6-TRICHLOROPHENOL	88-06-2	0.06	FDEP Leachability SCTL	0.020	0.333	0.133	0.030
2,4-DICHLOROPHENOL	120-83-2	0.003	FDEP Leachability SCTL	0.0010	0.333	0.133	0.030
2,4-DIMETHYLPHENOL	105-67-9	1.7	FDEP Leachability SCTL	0.57	1.33	0.667	0.333
2,4-DINITROPHENOL	51-28-5	0.06	FDEP Leachability SCTL	0.020	3.33	1.67	0.833
2,4-DINITROTOLUENE	121-14-2	0.0004	FDEP Leachability SCTL	0.0013	0.333	0.133	0.030
2,6-DINITROTOLUENE	606-20-2	0.0004	FDEP Leachability SCTL	0.0013	0.333	0.133	0.030
2-CHLORONAPHTHALENE	91-58-7	260	FDEP Leachability SCTL	87	0.333	0.167	0.0833
2-CHLOROPHENOL	95-57-8	0.7	FDEP Leachability SCTL	0.23	0.333	0.167	0.0833
2-METHYLNAPHTHALENE	91-57-6	8.5	FDEP Leachability SCTL	2.8	0.333	0.167	0.0833
2-METHYLPHENOL	95-48-7	0.3	FDEP Leachability SCTL	0.10	0.333	0.167	0.0833
2-NITROANILINE	88-74-4	0.1	FDEP Leachability SCTL	0.033	1.33	0.667	0.333
2-NITROPHENOL	88-75-5	NA	---	NA	0.333	0.133	0.030

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
2,2'-OXYBIS(1-CHLOROPROPANE)	108-60-1	0.009	FDEP Leachability SCTL	0.0030	0.333	0.133	0.030
3,3'-DICHLOROBENZIDINE	91-94-1	0.003	FDEP Leachability SCTL	0.0010	0.333	0.133	0.030
3-NITROANILINE	99-09-2	0.01	FDEP Leachability SCTL	0.0033	1.33	0.667	0.333
4,6-DINITRO-2-METHYLPHENOL	534-52-1	0.4	FDEP Leachability SCTL	0.13	3.33	1.67	0.833
4-BROMOPHENYL PHENYL ETHER	101-55-3	NA	---	NA	0.333	0.133	0.030
4-CHLOROPHENYL PHENYL ETHER	7005-72-3	NA	---	NA	0.333	0.133	0.030
4-CHLORO-3-METHYLPHENOL	59-50-7	0.4	FDEP Leachability SCTL	0.13	0.333	0.167	0.0833
4-CHLOROANILINE	106-47-8	0.2	FDEP Leachability SCTL	0.067	0.333	0.133	0.030
4-METHYLPHENOL	106-44-5	0.03	FDEP Leachability SCTL	0.010	0.333	0.133	0.030
4-NITROANILINE	100-01-6	0.008	FDEP Leachability SCTL	0.0027	1.33	0.667	0.333
4-NITROPHENOL	100-02-7	0.3	FDEP Leachability SCTL	0.10	1.33	0.667	0.333
ACENAPHTHENE	83-32-9	2.1	FDEP Leachability SCTL	0.70	0.333	0.167	0.0833
ACENAPHTHYLENE	208-96-8	27	FDEP Leachability SCTL	9.0	0.333	0.167	0.0833
ACETOPHENONE	98-86-2	3.8	FDEP Leachability SCTL	1.3	0.333	0.167	0.0833
ANTHRACENE	120-12-7	2,500	FDEP Leachability SCTL	830	0.333	0.167	0.0833

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
<b>ATRAZINE</b>	<b>1912-24-9</b>	<b>0.06</b>	<b>FDEP Leachability SCTL</b>	<b>0.020</b>	0.333	0.133	0.030
BENZALDEHYDE	100-52-7	4.8	FDEP Leachability SCTL	1.6	0.333	0.167	0.0833
<b>BENZO(A)ANTHRACENE</b>	<b>56-55-3</b>	<b>0.15</b>	<b>USEPA R-RSL</b>	<b>0.050</b>	0.333	0.133	0.030
BENZO(A)PYRENE (1)	50-32-8	0.1	FDEP Residential SCTL	0.033	0.067	0.033	0.017
<b>BENZO(B)FLUORANTHENE</b>	<b>205-99-2</b>	<b>0.15</b>	<b>USEPA R-RSL</b>	<b>0.050</b>	0.333	0.133	0.030
BENZO(G,H,I)PERYLENE	191-24-2	2,500	FDEP Residential SCTL	830	0.333	0.167	0.0833
BENZO(K)FLUORANTHENE	207-08-9	1.5	USEPA R-RSL	0.50	0.333	0.167	0.0833
BIS(2-CHLOROETHOXY)METHANE	111-91-1	63	FDEP Leachability SCTL	21	0.333	0.167	0.0833
<b>BIS(2-CHLOROETHYL)ETHER</b>	<b>111-44-4</b>	<b>0.0001</b>	<b>FDEP Leachability SCTL</b>	<b>0.000033</b>	0.333	0.133	0.030
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	72	FDEP Residential SCTL	24	0.333	0.167	0.0833
BUTYL BENZYL PHTHALATE	85-68-7	310	FDEP Leachability SCTL	100	0.333	0.167	0.0833
CAPROLACTAM	105-60-2	NA	---	NA	0.333	0.133	0.070
<b>CARBAZOLE</b>	<b>86-74-8</b>	<b>0.2</b>	<b>FDEP Leachability SCTL</b>	<b>0.067</b>	0.333	0.133	0.030
CHRYSENE	218-01-9	15	USEPA R-RSL	5.0	0.333	0.167	0.0833
DIBENZO(A,H)ANTHRACENE	53-70-3	0.015	USEPA R-RSL	0.0050	0.333	0.133	0.030
DIBENZOFURAN	132-64-9	15	FDEP Leachability SCTL	5.0	0.333	0.167	0.0833
DIETHYL PHTHALATE	84-66-2	86	FDEP Leachability SCTL	29	0.333	0.167	0.0833

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
DIMETHYL PHTHALATE	131-11-3	380	FDEP Leachability SCTL	130	0.333	0.167	0.0833
DI-N-BUTYL PHTHALATE	84-74-2	47	FDEP Leachability SCTL	16	0.333	0.167	0.0833
DI-N-OCTYL PHTHALATE	117-84-0	1,700	FDEP Residential SCTL	570	0.333	0.167	0.0833
FLUORANTHENE	206-44-0	1,200	FDEP Leachability SCTL	400	0.333	0.167	0.0833
FLUORENE	86-73-7	160	FDEP Leachability SCTL	53	0.333	0.167	0.0833
HEXACHLOROBENZENE	118-74-1	0.4	FDEP Residential SCTL	0.13	0.333	0.167	0.0833
HEXACHLOROBUTADIENE	87-68-3	1	FDEP Leachability SCTL	0.33	0.333	0.167	0.0833
HEXACHLOROCYCLOPENTADIENE	77-47-4	9.5	FDEP Residential SCTL	3.2	0.333	0.167	0.0833
<b>HEXACHLOROETHANE</b>	<b>67-72-1</b>	<b>0.2</b>	<b>FDEP Leachability SCTL</b>	<b>0.067</b>	0.333	0.133	0.030
<b>INDENO(1,2,3-CD)PYRENE</b>	<b>193-39-5</b>	<b>0.15</b>	<b>USEPA R-RSL</b>	<b>0.050</b>	0.333	0.133	0.030
<b>ISOPHORONE</b>	<b>78-59-1</b>	<b>0.2</b>	<b>FDEP Leachability SCTL</b>	<b>0.067</b>	0.333	0.133	0.030
NAPHTHALENE	91-20-3	1.2	FDEP Leachability SCTL	0.40	0.333	0.167	0.0833
<b>NITROBENZENE</b>	<b>98-95-3</b>	<b>0.02</b>	<b>FDEP Residential SCTL</b>	<b>0.067</b>	0.333	0.133	0.030
<b>N-NITROSO-DI-N-PROPYLAMINE</b>	<b>621-64-7</b>	<b>0.00005</b>	<b>FDEP Leachability SCTL</b>	<b>0.000017</b>	0.333	0.133	0.030
N-NITROSODIPHENYLAMINE	86-30-6	0.4	FDEP Leachability SCTL	0.13	0.333	0.167	0.0833

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
PENTACHLOROPHENOL	87-86-5	0.03	FDEP Leachability SCTL	0.010	1.33	0.667	0.333
PHENANTHRENE	85-01-8	250	FDEP Leachability SCTL	83	0.333	0.167	0.0833
PHENOL	108-95-2	0.05	FDEP Leachability SCTL	0.017	0.333	0.133	0.035
PYRENE	129-00-0	880	FDEP Leachability SCTL	290	0.333	0.167	0.0833

(1) 8270D Low Level SOP utilized for PAHs.

**Matrix: Surface Soil**

**15.2.3 Analytical Group: VOCs**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1,1,1-TRICHLOROETHANE	71-55-6	1.9	FDEP Leachability SCTL	0.63	0.005	0.002	0.001
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.001	FDEP Leachability SCTL	0.00033	0.005	0.002	0.001
1,1,2-TRICHLOROETHANE	79-00-5	0.03	FDEP Leachability SCTL	0.010	0.005	0.002	0.001
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	11,000	FDEP Leachability SCTL	3.700	0.005	0.002	0.001
1,1-DICHLOROETHANE	75-34-3	0.4	FDEP Leachability SCTL	0.13	0.005	0.002	0.001

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1,1-DICHLOROETHENE	75-35-4	0.06	FDEP Leachability SCTL	0.020	0.005	0.002	0.001
1,2,4-TRICHLOROBENZENE	120-82-1	5.3	FDEP Leachability SCTL	1.8	0.005	0.002	0.001
<b>1,2-DIBROMO-3-CHLOROPROPANE</b>	<b>96-12-8</b>	<b>0.001</b>	<b>FDEP Leachability SCTL</b>	<b>0.00033</b>	<b>0.005</b>	<b>0.002</b>	<b>0.001</b>
<b>1,2-DIBROMOETHANE</b>	<b>106-93-4</b>	<b>0.0001</b>	<b>FDEP Leachability SCTL</b>	<b>0.000033</b>	<b>0.005</b>	<b>0.002</b>	<b>0.001</b>
1,2-DICHLOROBENZENE	95-50-1	17	FDEP Leachability SCTL	5.7	0.005	0.002	0.001
1,2-DICHLOROETHANE	107-06-2	0.01	FDEP Leachability SCTL	0.0033	0.005	0.002	0.001
1,2-DICHLOROPROPANE	78-87-5	0.03	FDEP Leachability SCTL	0.010	0.005	0.002	0.001
1,3-DICHLOROBENZENE	541-73-1	7	FDEP Leachability SCTL	2.3	0.005	0.002	0.001
1,4-DICHLOROBENZENE	106-46-7	2.2	FDEP Leachability SCTL	0.73	0.005	0.002	0.001
2-BUTANONE	78-93-3	17	FDEP Leachability SCTL	5.7	0.02	0.01	0.005
2-HEXANONE	591-78-6	1.4	FDEP Leachability SCTL	0.47	0.02	0.01	0.005
4-METHYL-2-PENTANONE	108-10-1	2.6	FDEP Leachability SCTL	0.87	0.02	0.01	0.005
ACETONE	67-64-1	25	FDEP Leachability SCTL	8.3	0.02	0.01	0.005
BENZENE	71-43-2	0.007	FDEP Leachability SCTL	0.0023	0.005	0.002	0.001

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
<b>BROMODICHLOROMETHANE</b>	<b>75-27-4</b>	<b>0.004</b>	<b>FDEP Leachability SCTL</b>	<b>0.0013</b>	<b>0.005</b>	<b>0.002</b>	<b>0.001</b>
BROMOFORM	75-25-2	0.03	FDEP Leachability SCTL	0.010	0.005	0.002	0.001
BROMOMETHANE	74-83-9	0.05	FDEP Leachability SCTL	0.017	0.005	0.002	0.001
CARBON DISULFIDE	75-15-0	5.6	FDEP Leachability SCTL	1.8	0.005	0.002	0.001
CARBON TETRACHLORIDE	56-23-5	0.04	FDEP Leachability SCTL	0.013	0.005	0.002	0.001
CHLOROBENZENE	108-90-7	1.3	FDEP Leachability SCTL	0.43	0.005	0.002	0.001
<b>CHLORODIBROMOMETHANE</b>	<b>124-48-1</b>	<b>0.003</b>	<b>FDEP Leachability SCTL</b>	<b>0.0010</b>	<b>0.005</b>	<b>0.002</b>	<b>0.001</b>
CHLOROETHANE	75-00-3	0.06	FDEP Leachability SCTL	0.020	0.005	0.002	0.001
CHLOROFORM	67-66-3	0.4	FDEP Residential SCTL	0.13	0.005	0.002	0.001
CHLOROMETHANE	74-87-3	0.01	FDEP Leachability SCTL	0.0033	0.005	0.002	0.001
CIS-1,2-DICHLOROETHENE	156-59-2	0.4	FDEP Leachability SCTL	0.13	0.005	0.002	0.001
CIS-1,3-DICHLOROPROPENE	10061-01-5	NA	---	NA	0.005	0.002	0.001
CYCLOHEXANE	110-82-7	NA	---	NA	0.005	0.002	0.001
DICHLORODIFLUOROMETHANE	75-71-8	44	FDEP Leachability SCTL	15	0.005	0.002	0.001

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
ETHYLBENZENE	100-41-4	0.6	FDEP Leachability SCTL	0.20	0.005	0.002	0.001
ISOPROPYLBENZENE	98-82-8	0.2	FDEP Leachability SCTL	0.067	0.005	0.002	0.001
TOTAL XYLENES	1330-20-7	0.2	FDEP Leachability SCTL	0.067	0.01	0.005	0.003
METHYL ACETATE	79-20-9	16	FDEP Leachability SCTL	5.3	0.005	0.002	0.001
METHYL TERT-BUTYL ETHER	1634-04-4	0.09	FDEP Leachability SCTL	0.030	0.005	0.002	0.001
METHYLENE CHLORIDE	75-09-2	0.02	FDEP Leachability SCTL	0.0067	0.02	0.01	0.005
METHYL CYCLOHEXANE	108-87-2	NA	---	NA	0.005	0.002	0.001
STYRENE	100-42-5	3.6	FDEP Leachability SCTL	1.2	0.005	0.002	0.001
TETRACHLOROETHENE	127-18-4	0.03	FDEP Leachability SCTL	0.010	0.005	0.002	0.001
TOLUENE	108-88-3	0.5	FDEP Leachability SCTL	0.17	0.005	0.002	0.001
TRANS-1,2-DICHLOROETHENE	156-60-5	0.7	FDEP Leachability SCTL	0.23	0.005	0.002	0.001
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NA	---	NA	0.005	0.002	0.001
TRICHLOROETHENE	79-01-6	0.03	FDEP Leachability SCTL	0.010	0.005	0.002	0.001
TRICHLOROFLUOROMETHANE	75-69-4	33	FDEP Leachability SCTL	11	0.005	0.002	0.001
VINYL CHLORIDE	75-01-4	0.007	FDEP Leachability SCTL	0.0023	0.005	0.002	0.001

**Matrix: Surface Soil**

**15.2.4 Analytical Group: Total Recoverable Petroleum Hydrocarbons (TRPH)**

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
TRPH	NA	340	FDEP Leachability SCTL	110	23	11	6

**15.3 Matrix: Surface Water, Storm Sewer Water, and Sediment Pore Water**

**15.3.1 Analytical Group: VOCs**

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,1,1-TRICHLOROETHANE	71-55-6	270	FDEP Surface Water	90	1	0.5	0.25
1,1,2,2-TETRACHLOROETHANE	79-34-5	10.8	FDEP Surface Water	3.6	1	0.5	0.25
1,1,2-TRICHLOROETHANE	79-00-5	16	FDEP Surface Water	5.3	1	0.5	0.25
1,1,2-TRICHLOROTRIFLUOROETHANE	76-13-1	NA	---	NA	1	0.5	0.25
1,1-DICHLOROETHANE	75-34-3	NA	---	NA	1	0.5	0.25
1,1-DICHLOROETHENE	75-35-4	3.2	FDEP Surface Water	1.1	1	0.5	0.25
1,2,4-TRICHLOROBENZENE	120-82-1	23	FDEP Surface Water	7.7	1	0.5	0.25
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	NA	---	NA	1	0.5	0.25
1,2-DIBROMOETHANE	106-93-4	13	FDEP Surface Water	4.3	1	0.5	0.25
1,2-DICHLOROBENZENE	95-50-1	99	FDEP Surface Water	33	1	0.5	0.25
1,2-DICHLOROETHANE	107-06-2	37	FDEP Surface Water	12	1	0.5	0.25
1,2-DICHLOROPROPANE	78-87-5	14	FDEP Surface Water	4.7	1	0.5	0.25
1,3-DICHLOROBENZENE	541-73-1	85	FDEP Surface Water	28	1	0.5	0.25

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,4-DICHLOROENZENE	106-46-7	3	FDEP Surface Water	1.0	1	0.5	0.25
2-BUTANONE	78-93-3	120,000	FDEP Surface Water	40,000	10	5	2.5
2-HEXANONE	591-78-6	NA	---	NA	10	5	2.5
4-METHYL-2-PENTANONE	108-10-1	23,000	FDEP Surface Water	7,700	10	5	2.5
ACETONE	67-64-1	1,700	FDEP Surface Water	570	10	5	2.5
BENZENE	71-43-2	71.28	FDEP Surface Water	24	1	0.5	0.25
BROMODICHLOROMETHANE	75-27-4	22	FDEP Surface Water	7.3	1	0.5	0.25
BROMOFORM	75-25-2	360	FDEP Surface Water	120	1	0.5	0.25
BROMOMETHANE	74-83-9	35	FDEP Surface Water	12	1	0.5	0.25
CARBON DISULFIDE	75-15-0	110	FDEP Surface Water	37	1	0.5	0.25
CARBON TETRACHLORIDE	56-23-5	4.42	FDEP Surface Water	1.5	1	0.5	0.25
CHLOROBENZENE	108-90-7	17	FDEP Surface Water	5.7	1	0.5	0.25
CHLORODIBROMOMETHANE	124-48-1	34	FDEP Surface Water	11	1	0.5	0.25
CHLOROETHANE	75-00-3	NA	---	NA	1	0.5	0.25
CHLOROFORM	67-66-3	470.8	FDEP Surface Water	160	1	0.5	0.25
CHLOROMETHANE	74-87-3	470.8	FDEP Surface Water	160	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	NA	---	NA	1	0.5	0.25
CIS-1,3-DICHLOROPROPENE	10061-01-5	NA	---	NA	1	0.5	0.25
CYCLOHEXANE	110-82-7	NA	---	NA	1	0.5	0.25
DICHLORODIFLUOROMETHANE	75-71-8	NA	---	NA	1	0.5	0.25
ETHYLBENZENE	100-41-4	610	FDEP Surface Water	200	1	0.5	0.25
ISOPROPYLBENZENE	98-82-8	260	FDEP Surface Water	87	1	0.5	0.25
TOTAL XYLENES	1330-20-7	370	FDEP Surface Water	120	5	2.5	1

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
METHYL ACETATE	79-20-9	NA	---	NA	5	2.5	1
METHYL TERT-BUTYL ETHER	1634-04-4	34,000	FDEP Surface Water	11,000	1	0.5	0.25
METHYLENE CHLORIDE	75-09-2	1,580	FDEP Surface Water	530	3	1.5	1
METHYL CYCLOHEXANE	108-87-2	NA	---	NA	1	0.5	0.25
STYRENE	100-42-5	460	FDEP Surface Water	150	1	0.5	0.25
TETRACHLOROETHENE	127-18-4	8.85	FDEP Surface Water	3.0	1	0.5	0.25
TOLUENE	108-88-3	480	FDEP Surface Water	160	1	0.5	0.25
TRANS-1,2-DICHLOROETHENE	156-60-5	11,000	FDEP Surface Water	3,700	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NA	---	NA	1	0.5	0.25
TRICHLOROETHENE	79-01-6	80.7	FDEP Surface Water	27	1	0.5	0.25
TRICHLOROFUOROMETHANE	75-69-4	NA	---	NA	5	2.5	1
VINYL CHLORIDE	75-01-4	2.4	FDEP Surface Water	0.80	1	0.5	0.25

**Notes:**

CAS = Chemical Abstract Service  
 LOQ = Limit of Quantitation  
 LOD = Limit of Detection  
 ug/L = Micrograms per liter  
 mg/kg = Milligrams per kilogram  
 NA = limit or goal not available for this analyte

**Bolded rows indicate that the PAL is between the laboratory LOQ and LOD. The Project 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 Risk Assessment.**

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

**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		
Prepare Rough Draft UFP-SAP Work Plan & Appendices	Tetra Tech	02/22/10	04/21/10		
Submit Rough Draft SAP Work Plan & Appendices to Navy	Tetra Tech	04/22/10	05/10/10		
Navy Review	Navy	04/22/10	05/10/10	Rough Draft - SAP	4/21/10
Receive Comments/Comment Resolution	Tetra Tech	05/10/10	05/12/10		
Revise Draft SAP Work Plan & Appendices to Address Navy Comments	Tetra Tech	05/12/10	05/14/10		
Regulator Review	FDEP	05/14/10	06/04/10	Draft SAP	05/14/10
Receive Comments/Comment Resolution	Tetra Tech	06/04/10	06/07/10		
Prepare and Submit Final SAP Work Plan & Appendices to Navy	Tetra Tech	06/07/10	06/11/10		
<b>Final Navy Review</b>	Navy	06/11/10	06/14/10	Final SAP	06/14/10
Submit Navy Approved Final SAP Work Plan & Appendices to FDEP	Tetra Tech	06/14/10	06/18/10	Final Navy Approved SAP	06/18/10

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Mobilization and Field Investigation Event 1 – DPT and MIP Sampling	Tetra Tech	06/21/10	07/16/10		
Laboratory Analysis – Event 1 Mobile Laboratory	KB Labs	06/21/10	07/16/10		
Mobilization and Field Investigation Event 2a – Offshore Sampling	Tetra Tech	9/20/10	09/24/10		
Laboratory Analysis – Event 2a – Offshore Sampling	Empirical and Microbial Insights	09/25/10	10/25/10		
Data Validation	Tetra Tech	10/25/10	11/25/10		
Event 2b – Monitoring Well Installation and Sampling	Tetra Tech	11/1/10	11/21/10		
Event 2b – Laboratory Analysis	Empirical and Microbial Insights	11/22/10	12/22/10		
Data Validation	Tetra Tech	12/23/10	1/23/11		
Prepare Draft Human Health Risk Assessment (HHRA) Report – OU 3	Tetra Tech	TBD	TBD		*
Prepare Draft Ecological Risk Assessment (ERA) Report – OU 3	Tetra Tech	TBD	TBD		
Navy Review of Draft HHRA Report – OU 3	Navy	TBD	TBD		

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Navy Review of Draft ERA Report – OU 3	Navy	TBD	TBD		
Prepare Draft-Final RI Report	Tetra Tech	TBD	TBD		
Navy Review Draft-Final RI Report	Navy	TBD	TBD		
Prepare Final RI Report OU 3	Tetra Tech	TBD	TBD	Final RI Report OU 3	*
Submit Final RI Report OU 3 (includes HHRA and ERA)	Tetra Tech	TBD	TBD		
Prepare Draft-Final FS Addendum OU 3	Tetra Tech	TBD	TBD		
Navy Review Draft-Final FS Addendum OU 3	Navy	TBD	TBD		
Prepare Final FS Addendum OU 3	Tetra Tech	TBD	TBD		
Submit Final FS Addendum OU 3	Tetra Tech	TBD	TBD	Final FS Addendum OU 3	*

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

The descriptions below provide an overview of the sampling strategy and rationale for the RI Addendum field activities at OU 3.

A Triad approach is being utilized to evaluate the nature and extent of contamination at OU 3. The Triad is an approach to decision-making for hazardous waste site cleanup. The approach provides a framework for efficiently using real-time environmental sensors and tools in order to improve decision-making at contaminated sites. The term Triad represents three main elements: systematic project planning (SPP), dynamic work strategies, and innovative rapid sampling and analytical technologies.

This SAP provides the elements of the systematic project planning stage, including the involvement of site stakeholders in the decision making process, and the development of DQOs and a CSM. Dynamic work strategies are strategies that incorporate adaptable project activities to site conditions as new information becomes available while work is underway. This allows for optimization of the data collection effort to better eliminate uncertainties and to integrate the site data into the evaluation of potential site remedies. Rapid sampling techniques will be utilized to provide real-time delineation of VOCs in the subsurface at the site via use of the MIP system combined with DPT groundwater sampling and on-site mobile laboratory analysis.

The overall approach for the proposed activities is to evaluate the potential risks to site workers, potential future residents, and ecological receptors in the St. Johns River that are posed by the previously identified areas of soil and groundwater contamination at OU 3. The intent is to evaluate risk posed by the Operable Unit as a whole rather than focus solely on individual areas of contamination. The NAS Jacksonville Partnering Team has devised a multi-phased approach to investigate the potential risks posed. The planned investigation is to build off prior efforts and fill existing data gaps that will allow the NAS Jacksonville Partnering Team to fully evaluate appropriate site remedial strategies to manage the risks posed.

**17.1 SOIL SAMPLING DESIGN, LOCATIONS, AND RATIONALE**

Due to the complexity of OU 3, the Team has agreed that soils within the boundary of OU 3 will not be a focus of the investigation. Since OU 3 will remain industrial in use for the foreseeable future, soil investigations will be limited to the boundaries of OU 3 for the purpose of establishing LUCs. Details regarding the soil sampling program are provided below.

Figure 17-1 provides the location of soil samples to be collected along the OU 3 boundary. The soil samples will be collected from the shallow surface interval (0-2 feet) at approximately 200-foot intervals

along the existing boundary as shown on Figure 17-1. All surface soil samples will be tested for VOCs, taking care to collect VOC samples from the depth interval of 0.5 to 2 feet in accordance with FDEP SOPs. In addition, two locations (SS16 and SS17) in the vicinity of the Battery Shop (PSC 14) as shown on Figure 17-1 will be sampled for lead and two sample locations (SS1 and SS2) in the vicinity of Hangar 101S will also be analyzed for listed metals, SVOCs/Low Level PAHs, and TRPH. Metals constituent analyses are to be collected from the 0-0.5 foot interval, while SVOCs/Low Level PAHs and TRPH will be collected from the 0.5-2 foot interval as required by FDEP SOPs. All surface soil samples will be collected via FDEP SOPs and will be submitted to Empirical for analyses.

Data will be compared to FDEP Residential SCTLs for each constituent. If exceedances are noted, step out samples will be selected for analysis. The locations of step out samples will be the nearest practical location where a sample may be collected, but in general will be within 100 feet of the sample identified as having a Residential SCTL exceedance.

In the event step out samples are selected for analysis, the second phase soil sampling event will be conducted 60 days after receipt of analytical data.

## **17.2 MIP/DPT GROUNDWATER SAMPLING DESIGN, LOCATIONS, AND RATIONALE**

Extensive groundwater data has been collected at OU 3. However, data gaps do exist that will be evaluated via a groundwater sampling program. Depending on the location of the sample, the rationale for its collection varies. Investigation of Areas upgradient of Building 106 will be conducted to provide further delineation of potential upgradient sources of contamination. Sampling in Areas C and D will be conducted to evaluate current plume conditions and to provide data for updating of the CSM. Sampling in Area E will be conducted to re-delineate the current extent of Area E since Area E data is nearly a decade old. Sampling in downgradient areas of Areas F and G will be conducted to complete delineation of these downgradient areas and to evaluate the potential offshore migration of contaminated groundwater to the St. Johns River. This data in turn will be used to further refine the location of offshore pore and surface water sampling efforts.

MIP/DPT sampling will be supported by the on-site mobile laboratory. All water samples collected during this effort will be analyzed by the mobile laboratory, with the exception of split samples submitted to an off-site laboratory as a QA measure as described in Worksheet #18.

Figures 17-2 through 17-4 provide the locations for rapid field screening sample locations. These locations will first be sampled and evaluated using the MIP tool. The MIP tool will provide data continuously throughout the subsurface zone. Subsequently, DPT methods will be utilized to collect groundwater grab samples for mobile laboratory analysis of VOCs. The general approach to groundwater

sampling will be to conduct profiling by the collection of groundwater at 10-foot depth intervals from the top of the encountered aquifer to the bottom of the aquifer. Sampling depths and locations may be field modified to evaluate discrete intervals of interest identified in the MIP survey. In some cases, it is anticipated the MIP sampling method may not be capable of reaching depth of interest. As a result, data collection in these areas will be dependent on DPT techniques.

Based on data obtained, some sample locations shown on Figures 17-2 through 17-4 may be modified to a more strategic location, or may not be performed altogether. The rationale in general would suggest that if no contamination is found in the boring, no additional borings will be advanced in the direction where contamination is not present. Sample locations may be moved to another location where more appropriate data may be obtained.

### **17.3 GROUNDWATER MONITORING WELL DESIGN, LOCATIONS, AND RATIONALE**

After completion of the MIP/DPT sampling program, the Partnering Team will evaluate MIP/DPT data from all areas at OU 3 for the purpose of designing and constructing additional monitoring wells. The purpose of the monitoring wells will be to provide confirmation of the plume extent and to provide locations for future monitoring of natural attenuation processes and appropriate monitoring program.

Although final design and location details cannot be provided at this time, it is anticipated that wells will be installed in the following areas.

- Upgradient of Building 106 to verify the extent of what is believed to be an upgradient source of contamination. It is anticipated that five to six wells may be required in this area.
- Source area below Building 106. Two wells will be installed in the intermediate aquifer zone. Both wells will include 5-foot screen intervals, one screening the top of the second aquifer unit (approximately 25-30 feet bgs) and a second at the base of the second aquifer at approximately 60-65 feet bgs.
- Plume axis along Areas C and D to provide locations for groundwater modeling and to support monitoring for MNA parameters.
- Perpendicular to the axis of the Building 106 plume to provide contaminant mass flux information for modeling purposes and source area depletion evaluation.
- Areas F and G to monitor possible discharge to the St. Johns River and to provide potential future point of compliance wells.

It is estimated that approximately 15 wells may be installed as part of this effort.

#### **17.4 RIVER SAMPLING DESIGN, LOCATIONS, AND RATIONALE**

Two groundwater plumes have been identified during prior field efforts that have demonstrated a potential to impact the St. Johns River via direct groundwater to surface water migration. One plume located on the north side of OU 3 from Areas C and D is located in the second aquifer zone separated from the shallow aquifer interval (Figure 10-4). Sampling previously conducted via DPT methods from a barge have shown the plume may be discharging to the river near the Boat House in an area where deep dredging has compromised the clay unit separating the shallow and second aquifer zones (See Section 10.5). However, depth profiling sampling has shown contamination to decrease as the water upwells toward the river and biological processes within the organic rich sediment that backfilled the dredged area may be serving to degrade the VOCs before the contaminants reach the surface water of the St. Johns River. In order to confirm if this is the case, a sampling effort will be conducted to evaluate both pore water in sediments in the river bottom and surface water in the area where discharge would be occurring. A sampling grid shown on Figure 17-6 has been established in the discharge areas.

As described in Section 14.1, a Trident Probe will be used at each sample location to delineate the area where groundwater is discharging to surface water of the St. Johns River. Differences in temperature and conductivity measured by the probe sensors indicate where groundwater discharge is occurring. After the area of groundwater discharge has been determined using the temperature and conductivity sensor probe, the integral pore water sampler probe will be used to collect pore water samples two feet below the sediment surface and surface water samples one foot above the sediment surface in locations where groundwater discharge is occurring. Surface water and pore water samples will be tested in an on-site mobile laboratory for target VOC constituents.

In four to six representative locations where VOCs in the pore water exceed PALs, the VOC mass flux will be determined using the UltraSeep system to measure the seepage rate of discharging groundwater and to collect a pore water sample at the surface of the sediment for VOC analysis as described in Section 14.1.

The second groundwater plume that has been identified as posing a threat to the St. Johns River is located at Areas F and G (Figure 10-5). In this area of OU 3, there is no clay layer separating a shallow and second aquifer zone and, as a result, the shallow aquifer extends from the water table to a depth of approximately 60 feet bgs. Based on further delineation of the downgradient plume areas for Areas F and G, as described in Section 17.2, a sampling grid will be established offshore for the purpose of obtaining pore water and surface water samples via the Trident Probe. This area will also include the potential discharge area from the eastern storm sewer in Area G, which discharges in the same general area. Pore water and surface water samples will be collected at the points shown on Figure 17-7 as previously described and will be tested in the on-site mobile laboratory for VOCs. Similar to the northern

plume area, if results exceed PALs, an UltraSeep meter will be deployed to determine the contaminant mass flux into the surface water body.

## **17.5 STORM SEWER SAMPLING DESIGN, LOCATIONS, AND RATIONALE**

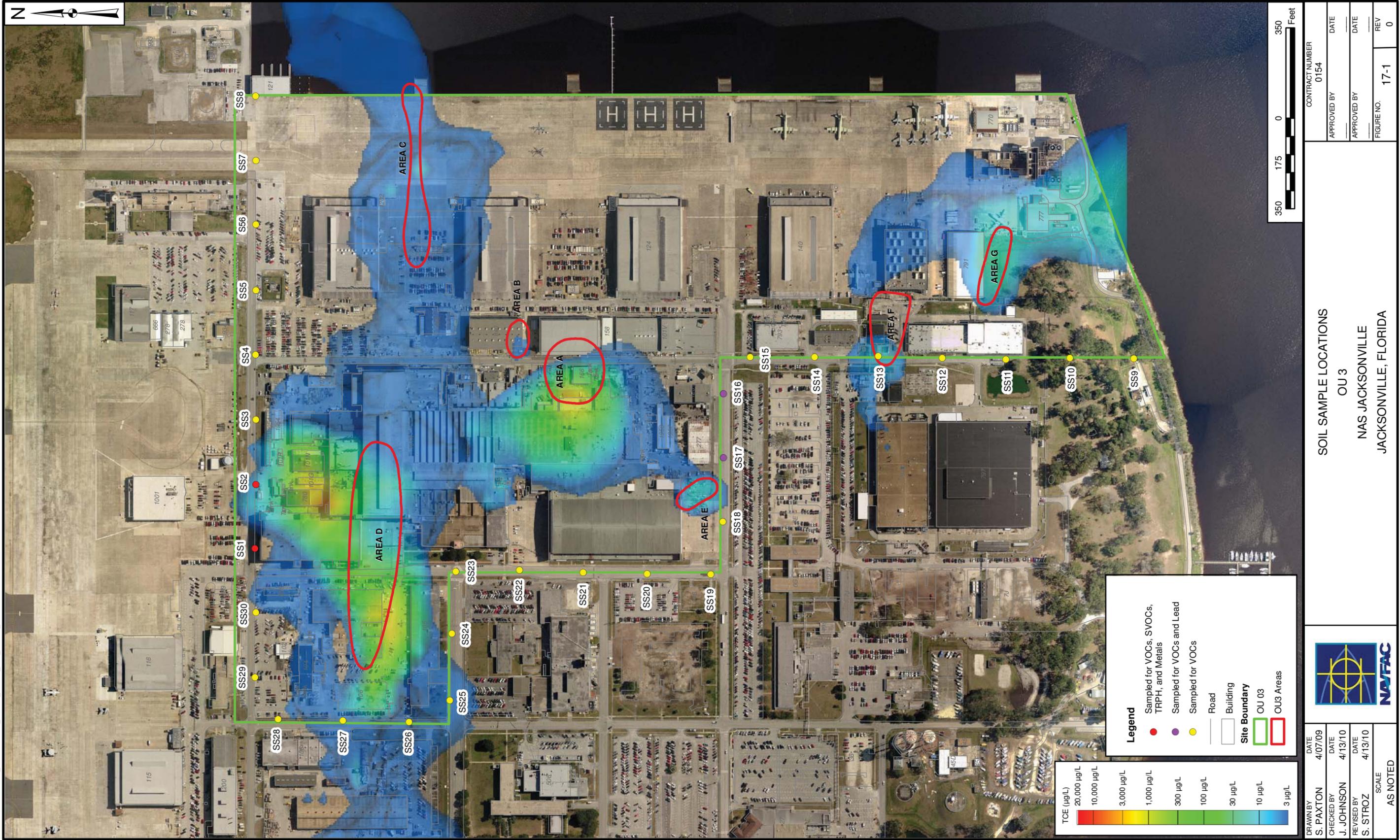
Contaminated groundwater entering storm sewers poses a potential risk to utility workers and ecological receptors in the St. Johns River. Data collected to date indicates that contaminated groundwater has been identified in two tidally influenced storm sewers with outfalls in the St. Johns River. One of these storm sewers, which is located within Areas A, F, and G, has been addressed in the prior ROD and a storm sewer monitoring program is in place. A second storm sewer was recently identified to be receiving contaminated groundwater discharges. This storm sewer is located in the downgradient areas of the Areas F and G groundwater plume, and the outfall for this storm sewer is approximately 100 feet offshore and is submerged beneath sediment in the St. Johns River (Figures 10-6 and 10-7). Dye testing conducted in the storm sewer indicates the storm sewer leaks and discharge may be encountered nearer to the shore than the actual designed outfall. Sampling conducted from accessible manholes and grated drains within the storm sewer has indicated that VOC constituents have been detected at levels exceeding Florida Marine SWCTLs during periods of low tide.

In order to determine if this discharge poses a potential risk to ecological receptors in the St. Johns River, a sampling event will be conducted on the storm sewer at the nearest accessible manhole or grated drain to the river over a full tidal cycle. Sampling will be conducted at one hour intervals from low tide to high tide and high tide to low tide and samples analyzed in the mobile laboratory for target VOCs. Storm sewer water samples will be collected via lowering a polyethylene sampling tubing into the water stream and pumping via a peristaltic pump, or by dipping a Teflon water sampling bucket into the water stream, whichever method is more practical based on field conditions in accordance with the SOPs listed in Worksheet #18. The location of the storm sewer sampling location is provided on Figure 17-5.

In addition, a survey will be completed to locate and sample any other storm sewers that discharge to the St. Johns River during dry periods. Storm sewers that discharge during dry periods are indicative of systems that are receiving infiltration of groundwater and therefore could be potentially impacted should contaminated groundwater be present. If any identified storm sewers are tidally influenced, sampling will be conducted at the outfall or closest manhole or grated drain if the outfall is submerged or otherwise inaccessible. Sampling will occur during low tide only and samples analyzed in the on-site mobile laboratory for target VOCs. If target VOCs are detected, additional storm sewer evaluation and sampling may be performed at a later date under a separate effort and a separate or updated SAP will be generated.

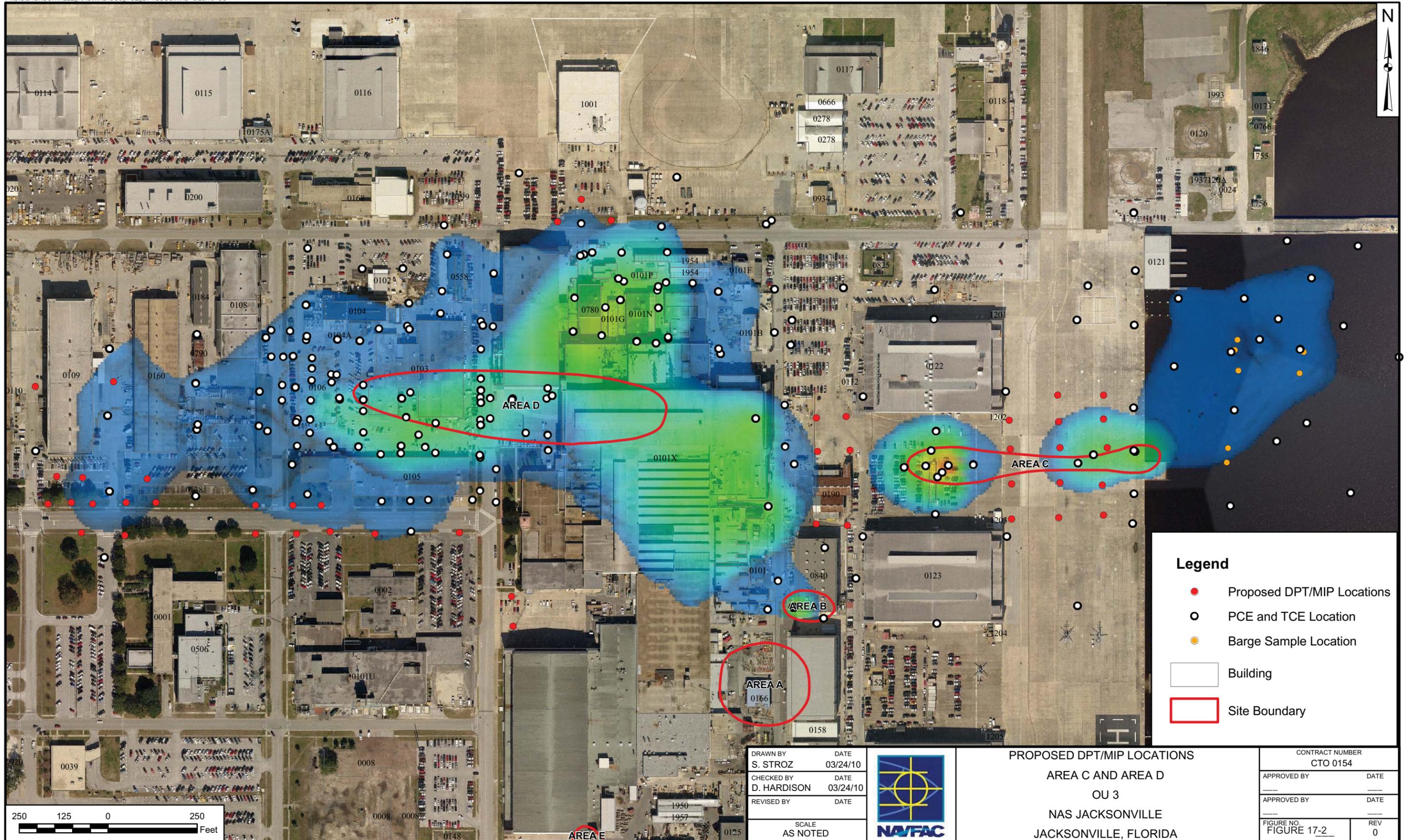
## **17.6 SAMPLING PROGRAM OPTIMIZATION**

The Triad approach allows for continual sample optimization through the review of near real-time data that allows for field decisions. MIP data will be used to identify areas of higher interest and lower interest so that DPT sampling can be modified to collect more valued data in high interest areas and limit data collection in low interest areas. In addition, use of the on-site mobile laboratory will allow the field teams to modify sample grids to eliminate samples in areas where contamination is not found to exist and to increase sampling density in areas where contamination is found. As a result, data produced is assured to be relevant and more complete, allowing for better decision making.



P:\GIS\JACKSONVILLE\_NAS\MD\OU3\_NORTH\_SOUTH\_TCE\_PLUME\_LOCS.MXD 4/13/10 SS

P:\GIS\JACKSONVILLE\_NAS\MXD\OU3\_TCE\_DPTLOCS.MXD 3/23/10 SS



**Legend**

- Proposed DPT/MIP Locations
- PCE and TCE Location
- Barge Sample Location
- Building
- Site Boundary

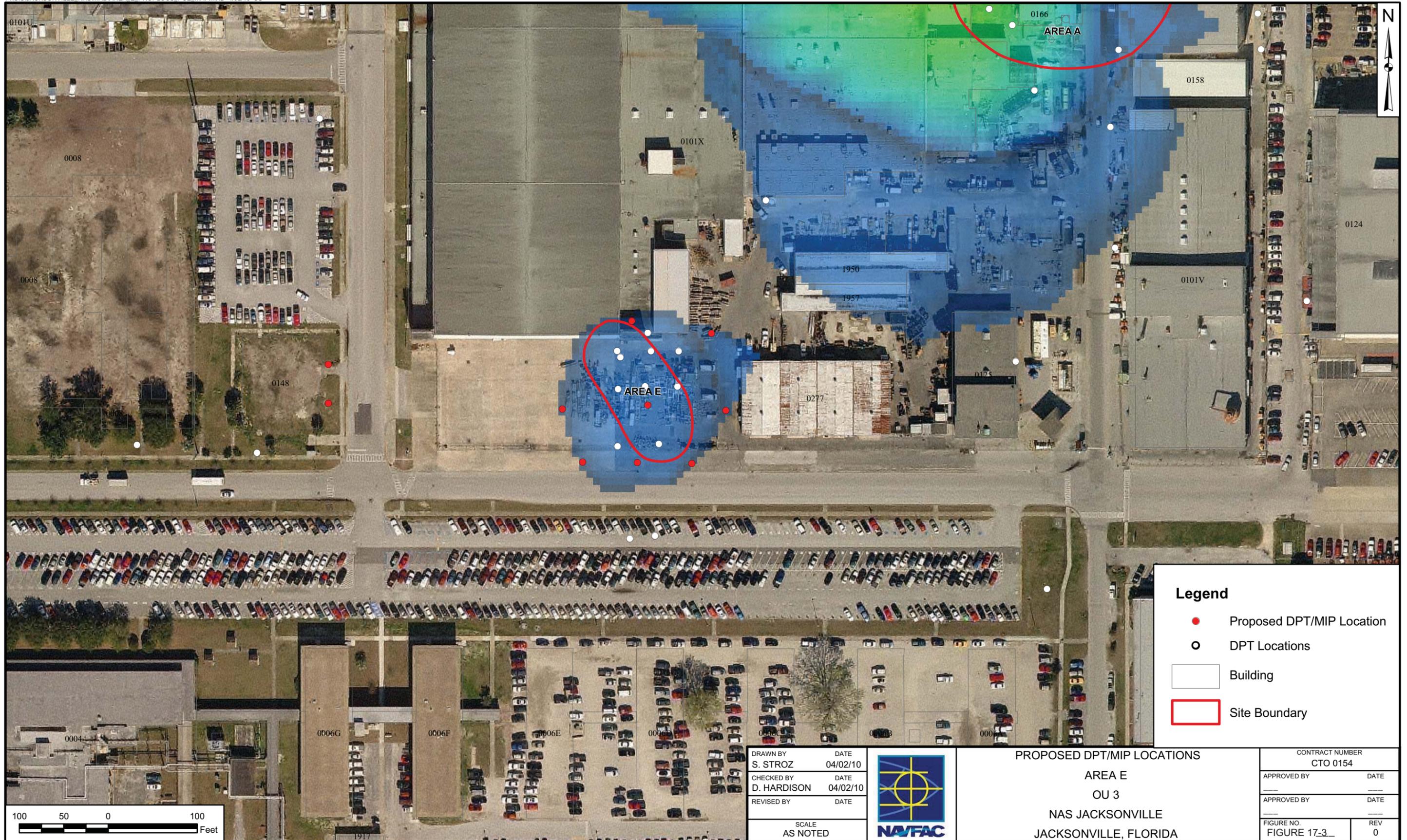
DRAWN BY S. STROZ	DATE 03/24/10
CHECKED BY D. HARDISON	DATE 03/24/10
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED DPT/MIP LOCATIONS  
 AREA C AND AREA D  
 OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

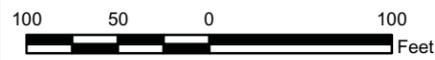
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FIGURE NO. FIGURE 17-2	REV 0

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**Legend**

- Proposed DPT/MIP Location
- DPT Locations
- Building
- Site Boundary



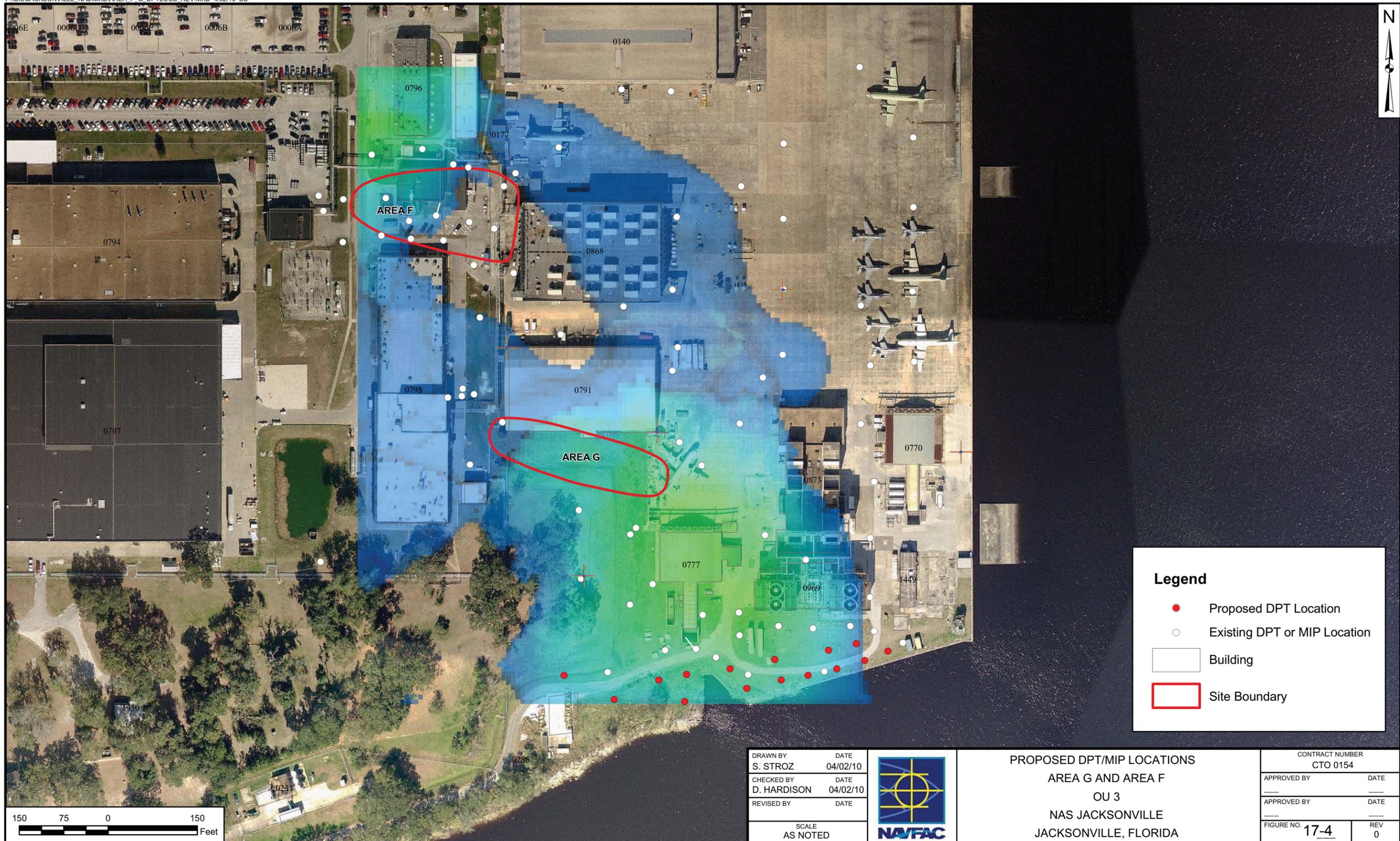
DRAWN BY S. STROZ	DATE 04/02/10
CHECKED BY D. HARDISON	DATE 04/02/10
REVISED BY	DATE
SCALE AS NOTED	



**PROPOSED DPT/MIP LOCATIONS**  
 AREA E  
 OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

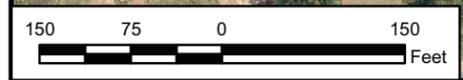
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FIGURE NO. FIGURE 17_3_	REV 0

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**Legend**

- Proposed DPT Location
- Existing DPT or MIP Location
- Building
- ▭ Site Boundary

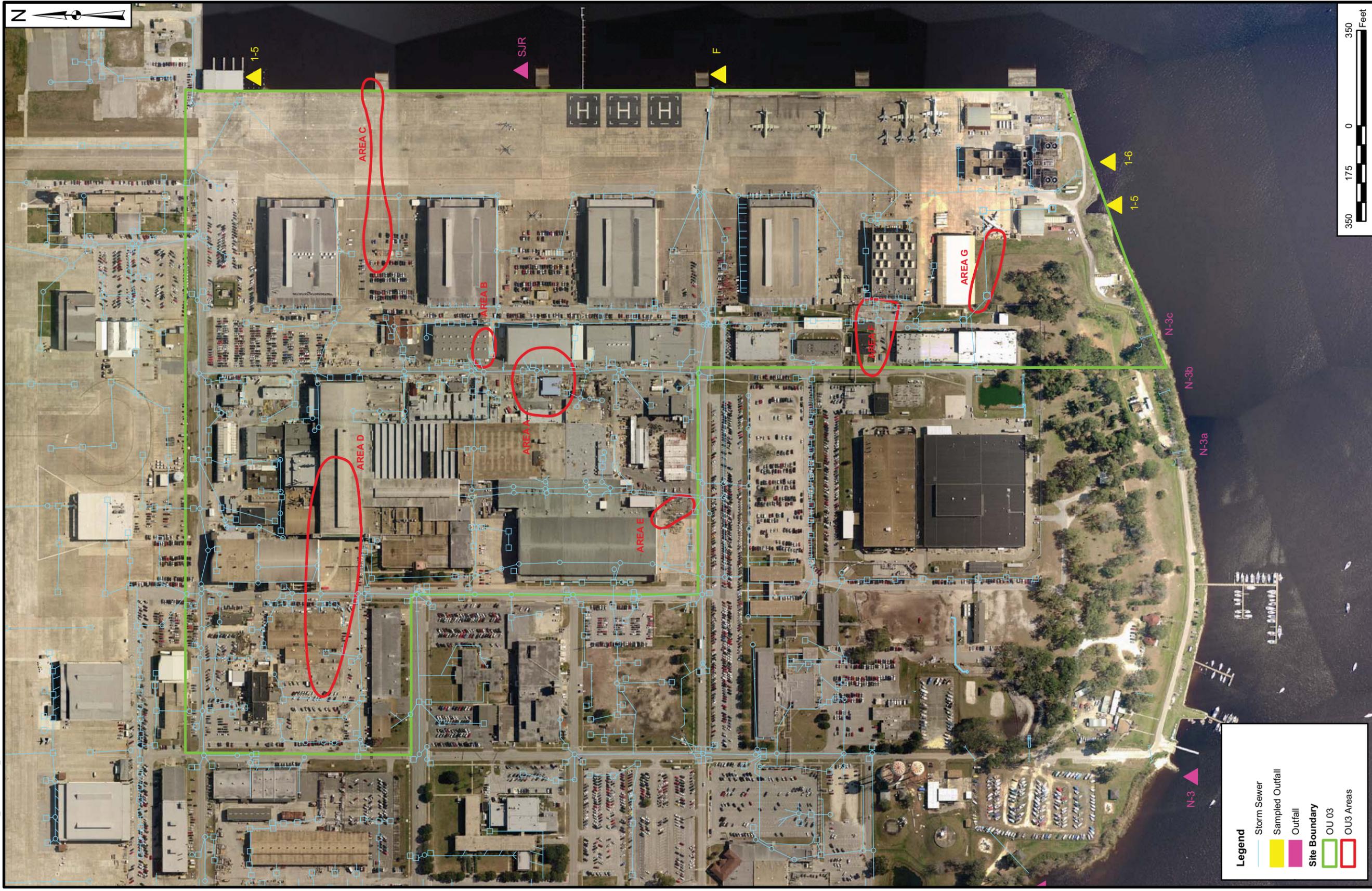


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CHECKED BY D. HARDISON	DATE 04/02/10
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED DPT/MIP LOCATIONS  
 AREA G AND AREA F  
 OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

CONTRACT NUMBER CTO 0154	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. <b>17-4</b>	REV 0



CONTRACT NUMBER		DATE	REV
APPROVED BY	DATE	DATE	0
APPROVED BY	DATE	DATE	0
FIGURE NO. FIGURE 17-5		REV	0

OU 3  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA



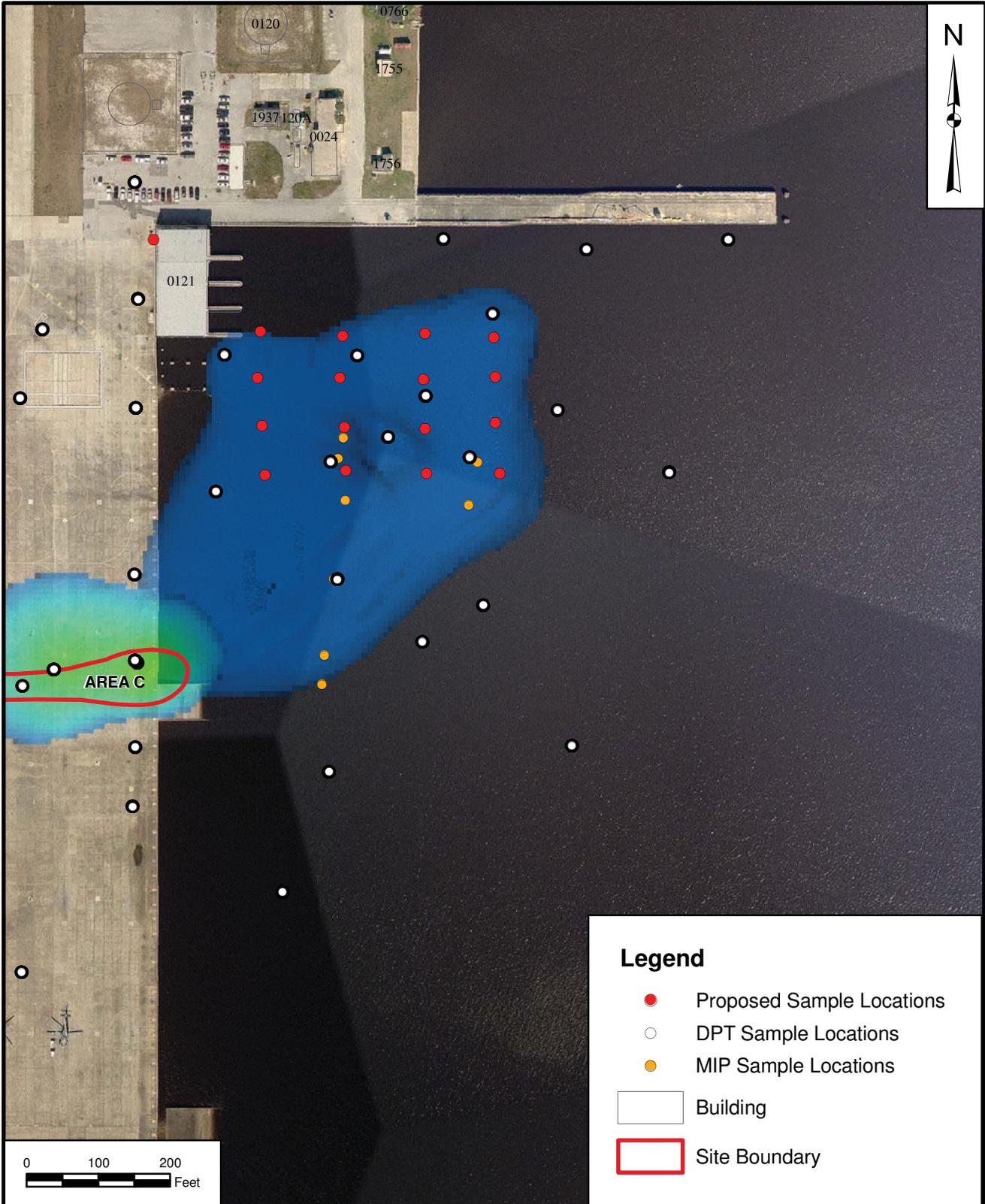
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S. STROZ	4/07/10	AS NOTED
CHECKED BY	DATE	
D. HARDISON	4/07/10	
REVISED BY	DATE	

**Legend**

- Storm Sewer
- Sampled Outfall
- Outfall
- Site Boundary
- OU 03
- OU3 Areas

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P:\GIS\JACKSONVILLE\_NAS\MXD\AREA\_C\_CONTAMINENTS.MXD 04/09/10 SS



**Legend**

- Proposed Sample Locations
- DPT Sample Locations
- MIP Sample Locations
- Building
- Site Boundary

DRAWN BY S. STROZ	DATE 04/07/10
CHECKED BY D. HARDISON	DATE 04/09/10
REVISED BY	DATE
SCALE AS NOTED	



TRIDENT PROBE SAMPLE LOCATIONS  
 AREA C  
 NAS JACKSONVILLE  
 JACKSONVILLE, FLORIDA

CONTRACT NUMBER 0154	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. FIGURE 17-6	REV 0



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

The Triad approach allows for continual sampling optimization. As a result, total sample numbers and depths are to be determined for MIP/DPT sampling and pore water sampling efforts. Split samples will be analyzed in the fixed-base laboratory on a minimum of 5 percent of the VOC samples that are analyzed in the on-site laboratory for each environmental medium (MIP/DPT groundwater, surface water, sediment pore water, and storm sewer water). Field duplicates for all media will be collected on a minimum of 5 percent of the total samples for analysis. Sample designation protocol is as follows:

**Surface Soil:** Station ID, Operable Unit ID, Surface Soil ID, depth interval, month, and year.

Example: JAX-OU3-SS1-0.5-2'-06/2010

**MIP/DPT Groundwater:** Station ID, Operable Unit ID, Boring ID, depth (screen) interval, month, and year.

Example: JAX-OU3-B1-24-28-06/2010

**Storm Sewer:** Station ID, Operable Unit ID, Manhole #, depth interval, time, day, month, and year.

Example: JAX-OU3-MH1-4.5'-10:20-21/06/2010

**Pore Water/Surface Water:** Station ID, Operable Unit ID, Sediment Location #, Sample Type (Groundwater Pore Water/Surface Water Pore Water/Surface Water), depth, month, and year.

Example: JAX-OU3-SD1-GWPW-1.5'-06/2010

**New Monitoring Wells:** Station ID, Operable Unit ID, Area ID, Well #, bottom of the screened interval, month, and year.

Example: JAX-OU3-AreaG-MW21-25'-06/2010

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
<b>Surface Soil Sample Locations</b>							
JAX-OU3-SS1-0-0.5'- mm/yyyy	Soil (surface)	NA	NA	Metals	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS1-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs, SVOCs/ Low Level PAHs, TRPH	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS2-0-0.5'- mm/yyyy	Soil (surface)	NA	NA	Metals	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS2-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs, SVOCs/ Low Level PAHs, TRPH	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS3-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS4-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS5-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS6-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS7-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS8-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS9-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS10-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS11-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS12-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS13-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS14-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS15-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS16-0-0.5'- mm/yyyy	Soil (surface)	NA	NA	Lead Only	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS16-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS17-0-0.5'- mm/yyyy	Soil (surface)	NA	NA	Lead Only	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS17-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS18-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS19-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS20-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS21-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS22-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS23-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS24-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS25-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS26-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
JAX-OU3-SS27-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS28-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS29-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination
JAX-OU3-SS30-0.5-2'- mm/yyyy	Soil (surface)	NA	NA	VOCs	5%	FC1000, FD1000, FM1000,FQ1000, FS1000, FS3000 SA-1.3, GH-1.5, SA-6.3, SA-7.1, SA-6.1, CT-04	LUC Boundary determination

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth <sup>2</sup> (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
<b>DPT/MIP Sample Locations</b>							
JAX-OU3-BX-xx-xx'- mm/yyyy	Groundwater (10' intervals from top of aquifer to bottom)	NA	NA	VOCs (Screening)	5%	FC1000, FD1000, FM1000, FQ1000, FS1000, FS2000, FS2200, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, SA-1.2, SA-1.3, GH-1.5, SA-2.5, SA-6.3, SA-7.1, SA-1.1, SA-6.1, CT-04	Plume Delineation
<b>Note:</b> Proposing approximately 58 DPT locations within OU 3. Boring numbers will be number sequentially starting with B1, B2, B3, etc.							
<b>Storm Sewer Sample Locations</b>							
JAX-OU3,MHX-X.X'- hr:min-dd/mm/yyyy	Water	Yes	NA	VOCs	5%	FC1000, FD1000, FM1000, FQ1000, FS1000, FS2000, FS2100, FT1000, SA-1.3, GH-1.5, SA-2.5, SA-6.3, SA-7.1, SA-1.1, SA-6.1, CT-04	Contaminant Detection
<b>Note:</b> Storm sewer location is associated with Area G, Storm Sewer 1-6 as shown on Figure 17-5. Other storm sewers/manholes may be sampled based on site survey.							

Sampling Location/ ID Number	Matrix	Synoptic Water Level	Well Depth (feet)	Analytical Group	Number of Field Duplicates	Sampling SOP Reference	Rationale for Sampling Location
<b>Pore Water/Surface Water &amp; Groundwater Sample Locations</b>							
JAX-OU3-SDX-GWPW- X.X'-mm/yyyy	water	NA	NA	VOCs	5%	FC1000, FD1000, FM1000, FQ1000, FS1000, FS2000, FS2100, FS2200, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, SA-1.3, GH-1.5, SA-2.5, SA-6.3, SA-7.1, SA-1.1, SA-6.1, CT-04	Contaminant Delineation
<b>Note:</b> Number samples sequentially starting with SD1, SD2, SD3, etc. GW = groundwater, PW = pore water, SW = surface water. GW and SW will be determined in the field from results of field testing. Anticipate 36 sample locations.							
<b>Monitoring Well Samples</b>							
JAX-OU3-AreaX- MWXX-XX'-mm/yyyy	Water	Yes	TBD	VOCs, MNA Parameters	5%	FC1000, FD1000, FM1000, FQ1000, FS1000, FS2000, FS2200, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600, SA-1.3, GH-1.2, GH-1.5, GH-2.8, SA-2.5, SA-6.3, SA-7.1, SA-1.1, SA-6.1, CT-04	Monitoring
<b>Note:</b> Monitoring well locations are determined through the Triad approach and location IDs will be determined according to location of installation at the time of installation.  MNA parameter analyses are shown in Worksheet #19.							

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

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference <sup>1</sup>	Containers (number, size, and type) <sup>2</sup>	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis) <sup>3</sup>
Groundwater and Aqueous QC Blanks	VOCs	SW-846 5030/8260B, Empirical SOP-202	Three - 40 milliliter (mL) glass vials	5 mL	Hydrochloric acid (HCl) to pH<2; Cool to 4 (± 2) °C; no headspace	14 days to analysis
	SVOCs and Low Level PAHs	SW-846 3510C/3520/8270D, Empirical SOP-201//300	Two 1 - liter (L) glass amber bottles	1000 mL	Cool to 4 (± 2) °C	7 days until extraction, 40 days to analysis
	TAL Metals (Including Mercury) and Molybdenum, Total and Dissolved Iron, and Lead	SW-846 3010A/6010C/7470A, Empirical SOP-100/103/105	One - 500 mL plastic bottle	50 mL / 30 mL mercury	Nitric acid (HNO <sub>3</sub> ) to pH <2; Cool to 4 (± 2) °C	180 days to analysis except mercury; 28 days to analysis for mercury
	MNA Parameters					
	Anions (nitrate, nitrite, chloride, and sulfate)	EPA 300.0, Empirical SOP-145	One - 500 mL plastic bottle	5 mL for each analyte	Cool to 4 (± 2) °C	Nitrate/Nitrite - 48 hours to analysis; Chloride/ Sulfate - 28 days to analysis
	Alkalinity	SM 2320B, Empirical SOP-154	One 500-mL plastic bottle	25 mL	Cool to 4 (± 2) °C	14 days to analysis
	Dissolved Sulfide	SM4500S F, Empirical SOP-153	One - 500 mL plastic bottle	200 mL	1 mL 2 Normal zinc acetate with Sodium hydroxide (NaOH) to a pH >9; Cool to 4 (± 2) °C	7 days to analysis
	TOC	SM 5310C, Empirical SOP-221	One - 500 mL plastic bottle	250 mL	Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ) to pH <2; Cool to 4 (± 2) °C	28 days to analysis
	Dissolved Gases (methane, ethane, ethene)	RSK SOP 175, Empirical SOP-236	Three - 40 mL glass vials	15 mL	HCl to pH <2; Cool to 4 (± 2) °C	14 days to analysis
Groundwater/ Sediment Pore Water	Dehalococoides and reductase genes	Lab Proprietary Methods, MI SOP DNA-qPCR, MI SOP DNA Ext,	One - 1L plastic	1 L	Cool to 4 (± 2) °C	Extract within 48 hours and freeze at < -10°C until analysis

<b>Matrix</b>	<b>Analytical Group</b>	<b>Analytical and Preparation Method / SOP Reference<sup>1</sup></b>	<b>Containers (number, size, and type)<sup>2</sup></b>	<b>Sample Volume (units)</b>	<b>Preservation Requirements (chemical, temperature, light protected)</b>	<b>Maximum Holding Time (preparation/analysis)<sup>3</sup></b>
Soil	VOCs	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram Encore samplers or terracores	5 grams	2x40mL in water and 1x40mL in methanol, freeze to < -10 °C	48 hours from sampling to preservation, 14 days to analysis
	SVOCs and Low Level PAHs	SW-846 3546/8270C/ Empirical SOP-201/343	One 4-ounce glass jar	30 grams	Cool to 4 (± 2) °C	14 days until extraction, 40 days to analysis
	TAL Metals (Including Mercury) and Molybdenum, and Lead Only	SW-846 3050B/ 6010C/7471A Empirical SOP-100/104/105	One 4-ounce glass jar	1 to 2 grams / 0.3 gram for mercury	Cool to 4 (± 2) °C	180 days to analysis except mercury, 28 days to analysis for mercury
	TRPH	FL-PRO Empirical SOP-338	One 4-ounce glass jar	30 grams	Cool to 4 (± 2) °C	14 days to analysis
Groundwater, Storm Sewer Water, Sediment Pore Water, and Surface Water	VOCs Screening Level Data	SW-846 8260B KB SOP01VOC	Two - 40 mL glass vials	5 mL	HCl to pH<2; Cool to 4 (± 2) °C; no headspace	14 days to analysis

Notes:

- <sup>1</sup> 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.
- <sup>2</sup> 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.
- <sup>3</sup> Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

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

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs <sup>1</sup>	Number of Field Blanks	Number of Equipment Blanks	Number of VOC Trip Blanks	Number of PT <sup>2</sup> Samples	Total Number of Samples to Lab
Surface Soil	VOCs	30	2	2/2	0	0	2	0	34
	SVOCs/ PAHs	2	1	0/0	0	0	NA	0	3
	Metals	2	1	0/0	0	0	NA	0	3
	Lead Only	2	1	0/0	0	0	NA	0	3
	TRPH	2	1	0/0	0	0	NA	0	3
Groundwater (MIP/DPT Samples)	VOCs (On-Site Screening)	58	3	3/3	0	0	0	0	61
Storm Sewer Water	VOCs (On-Site Screening)	6	1	1/1	0	1	1	0	9
Surface and Sediment Pore Water	VOCs (On-Site Screening)	36	2	2/2	0	2	2	0	42
	Dehalococoides and reductase genes (Sediment Pore Water)	12	0	0/0	0	0	NA	0	12

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs <sup>1</sup>	Number of Field Blanks	Number of Equipment Blanks	Number of VOC Trip Blanks	Number of PT <sup>2</sup> Samples	Total Number of Samples to Lab
Off-Site Analysis for Confirmation of On-site Analytical Data (5% minimum)	VOCs	10	1	1/1	0	0	3	0	14
Groundwater (Monitoring Wells) <sup>3</sup>	VOCs	15	1	1/1	0	1	2	0	19
	SVOCs/ Low Level PAHs	15	1	1/1	0	0	NA	0	16
	Metals	15	1	1/1	0	1	NA	0	17
	MNA Parameters								
	Iron and Manganese (Total and Dissolved)	15	0	0/0	0	0	NA	0	15
	Dissolved Gases	15	0	0/0	0	0	NA	0	15
	TOC	15	0	0/0	0	0	NA	0	15
	Anions	15	0	0/0	0	0	NA	0	15
	Dissolved Sulfide	15	0	0/0	0	0	NA	0	15
	Alkalinity	15	0	0/0	0	0	NA	0	15

Matrix	Analytical Group	Number of Sampling Locations	Number of Field Duplicates	Number of MS/MSDs <sup>1</sup>	Number of Field Blanks	Number of Equipment Blanks	Number of VOC Trip Blanks	Number of PT <sup>2</sup> Samples	Total Number of Samples to Lab
	Dehalococcoides and reductase genes	15	0	0/0	0	0	NA	0	15

Notes:

- <sup>1</sup> Although the matrix spike/matrix spike duplicate (MS/MSD) is not typically considered a field QC, it is included here because location determination is often established in the field. The MS/MSD are not included in the Total Number of Samples to the Lab. For Metals, a laboratory duplicate will be collected in place of an MSD.
- <sup>2</sup> PT = Proficiency Testing (previously known as performance evaluation sample)
- <sup>3</sup> The quantity of monitoring wells will be determined based on the results of the initial field activities; however, 15 monitoring wells will be assumed in this table for planning purposes.

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

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP <sup>1</sup>	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature, Revision 2, March 2009	Tetra Tech	NA	Y	Sample identification will follow the logic outlined in Worksheet #18. Contained in Appendix A.
CT-05	Database Records and Quality Assurance, Revision 2, January 2001	Tetra Tech	NA	N	Contained in Appendix A.
FC 1000	Cleaning/Decontamination Procedures, December 2008	FDEP	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized [DI] water)	N	Contained in Appendix A.
FD 1000	Documentation Procedures, December 2008	FDEP	Documentation of all sampling activities (log book, sampling logs, chain-of-custodies)	N	Contained in Appendix A.
FM 1000	Field Planning and Mobilization, December 2008	FDEP	Equipment supply and preparation and assemble field record supplies.	N	Contained in Appendix A.
FQ 1000	Field Quality Control Requirements, December 2008	FDEP	NA	N	Contained in Appendix A.

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP <sup>1</sup>	Equipment Type	Modified for Project Work? (Y/N)	Comments
FS 1000	General Sampling Procedures, December 2008	FDEP	NA	N	Contained in Appendix A.
FS 2000	General Aqueous Sampling, December 2008	FDEP	NA	N	Contained in Appendix A.
FS 2100	Surface Water Sampling, December 2008	FDEP	NA	N	Contained in Appendix A.
FS 2200	Groundwater Sampling, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FS 2212	Well Purging Techniques, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FS 3000	Soil Sampling, December 2008	FDEP	NA	N	Contained in Appendix A.
FT 1000	Field Testing General, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1100	Field pH, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1200	Field Specific Conductance, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP <sup>1</sup>	Equipment Type	Modified for Project Work? (Y/N)	Comments
FT 1300	Field Salinity, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1400	Field Temperature, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1500	Field Dissolved Oxygen, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
FT 1600	Field Turbidity, December 2008	FDEP	Turbidity meter, such as LaMotte Model 2008, or similar	N	Contained in Appendix A.
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement, Revision 2, September 2003	Tetra Tech	Electronic water level indicator	N	Contained in Appendix A.
GH-1.5	Borehole and Sample Logging, Revision 1, June 1999	Tetra Tech	NA	N	Contained in Appendix A.
SA-1.1	Site Reconnaissance, Revision 7, April 2008	Tetra Tech	Safety equipment, maps, geologic tools, monitoring equipment, marking items, and field notebooks	N	Contained in Appendix A.

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP <sup>1</sup>	Equipment Type	Modified for Project Work? (Y/N)	Comments
SA-1.2	Surface Water and Sediment Sampling, Revision 5, March 2008	Tetra Tech	Peristaltic pump, bailer, dip sampler, weighted bottle, hand pump Kemmerer, depth-integrating sampler	N	Contained in Appendix A.
SA 1.3	Soil and Rock Drilling Methods, Revision 9, April 2008	Tetra Tech	NA	N	Contained in Appendix A.
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™), Revision 3, September 2003	Tetra Tech	Sampling kit, macrocore sampler, probe sampling adapters, roto hammer with bit	N	Contained in Appendix A.
SA-6.1	Non-Radiological Sample Handling Revision 3, February 2004	Tetra Tech	Sample Bottle Ware, Packaging Material, Shipping Materials	N	Contained in Appendix A.
SA-6.3	Field Documentation Revision 3, March 2009	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	Contained in Appendix A.
SA-7.1	Decontamination of Field Equipment, Revision 6, January 2009	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, DI water)	N	Contained in Appendix A.

Notes:

<sup>1</sup> FDEP Field SOPs can be obtained at the following website: <http://www.dep.state.fl.us/labs/qa/sops.htm>

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

Field Equipment	Activity <sup>1</sup>	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference <sup>2,3</sup>	Comments
Electric 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
YSI 600 Series (or similar) Multi-Parameter 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	FDEP FT 1000 through 1500 and Manufacturer's Guidance	None
LaMotte Model 2008 (or similar) Turbidity Meter	Visual Inspection  Calibration/Verification	Daily  Beginning and end of day	RPD of $\pm 10\%$ (6 measurements of 2 successive samples of a 20 NTU standard)  Accuracy of $\pm 10\%$ at 20 NTU (Mean of the measured values must be 18-22 NTU)	Operator correction or replacement	Tetra Tech FOL or designee	FDEP FT 1600, Field Measurement of Turbidity and Manufacturer's Guidance	If an acceptable turbidity meter model is not used, submittal of an Alternate Test Procedure application is required

Notes:

- <sup>1</sup> Activities may include: calibration, verification, testing, maintenance, and/or inspection.
- <sup>2</sup> Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).
- <sup>3</sup> FDEP Field SOPs can be obtained at the following website: <http://www.dep.state.fl.us/labs/qa/sops.htm>

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

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
Empirical SOP-100	Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C (Revision 19, 04/20/09)	Definitive	Groundwater, Soil, and Aqueous QC Blanks/ Metals digestion	NA/ Preparation	Empirical	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique (Revision 18, 04/11/10)	Definitive	Groundwater/ Metals	Cold-vapor atomic absorption (CVAA)	Empirical	N
Empirical SOP-104	Mercury Analysis in Soil/sediment by Manual Cold Vapor Technique (Revision 19, 04/11/10)	Definitive	Soil/ Metals	CVAA	Empirical	N
Empirical SOP-105	Metals Analysis by ICP Technique Methods 200.7, SW846 6010B, SM 19 <sup>th</sup> Edition 2340B, USEPA ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Groundwater, Soil, and Aqueous QC Blanks/ Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Empirical	N
Empirical SOP-145	Determination of Inorganic Anions in Water by Ion Chromatography using Dionex DX-500 Ion Chromatographs with Hydroxide Eluent and Dionex Column AS18, Method 300.0 Guidance (Revision 7, 03/25/10)	Definitive	Groundwater / Anions	Dionex Ion Chromatography (IC)	Empirical	N
Empirical SOP-153	Sulfide Method 376.1 and Standard Methods SM4500S-F(19 <sup>th</sup> ED) Titrametric, Iodine) with Sample Pretreatment to Remove Interfering Substances or to Concentrate the Sulfide (Revision 3, 05/27/09)	Definitive	Groundwater / Dissolved Sulfide	Buret	Empirical	N
Empirical SOP-154	Alkalinity by EPA Method 310.1, SM2320B (Revision 5, 05/27/09)	Definitive	Groundwater/ Alkalinity	Buret/ pH meter	Empirical	N
Empirical SOP-201	GC/MS Semivolatiles by EPA Method 625 and SW846 Method 8270C and 8270D Including Additional Appendix IX Compounds (Revision 19, 04/11/10)	Definitive	Groundwater and Soil/ SVOCs and PAHs	Gas Chromatography/ Mass Spectroscopy (GC/MS)	Empirical	N
Empirical SOP-202	GC/MS Volatiles by Method 624 and SW846 Method 8260B (Revision 22, 09/30/09)	Definitive	Groundwater, Soil, and Aqueous QC Blanks/ VOCs	GC/MS	Empirical	N
Empirical SOP-221	Total Organic Carbon SM5310C, USEPA Method 415.1 and SW846 Method 9060/9060A and Lloyd Kahn Method (Revision 8, 04/28/09)	Definitive	Groundwater/ TOC	TOC Analyzer	Empirical	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
Empirical SOP-236	Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175 (Automated Headspace) (Revision 1, 04/28/09)	Definitive	Groundwater/ Dissolved Gases	GC/MS	Empirical	N
Empirical SOP-343	BNA & Pesticide/PCBs & TPH Non-Aqueous Matrix (Microwave Extraction) Using SW-846 Method 3546 (Revision 17, 09/23/08)	Definitive	Soil/ SVOCs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-300	GC/MS- Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis (Revision 17, 09/23/08)	Definitive	Groundwater / SVOC/PAHs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-338	FL-PRO ( Extractable Petroleum Hydrocarbons) Aqueous and Solid Matrix (Revision 7, 02/24/10)	Definitive	Groundwater, Soil, and Aqueous QC Blanks/TRPH	GC/FID	Empirical	N
Empirical SOP-404	Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures (Revision 13, 06/29/09)	NA	Log-in	NA/ Log-in	Empirical	N
Empirical SOP-405	Analytical Laboratory Waste Disposal (Revision 5, 06/23/09)	NA	Disposal	NA/ Disposal	Empirical	N
Empirical SOP-410	Standard Operating Procedures for Laboratory Sample Storage, Secure Areas, and Sample Custody (Revision 7, 06/23/09)	NA	Log-in	NA/ Log-in	Empirical	N
MI SOP-DNA EXT	Extraction of DNA from Environmental Samples (Matrix-Water, Soil, Biofilm, Bio-Sep beads) (Revision 1, 01/05/06)	Screening	Groundwater/ DNA Extraction	Incubator	Microbial Insights	N
MI SOP-DNA qPCR	Quantitative Polymerase Chain Reaction (qPCR) (Revision 1, 01/05/06)	Screening	Groundwater/ Dehalococcoides and reductase genes	Applied Biosystems	Microbial Insights	N
MI SOP-SAMREC	Sample Receiving (Revision 1.1, 11/14/08)	NA	Sample Receiving	NA/ Sample Receiving	Microbial Insights	N
MI SOP-WASTE DISPOSAL	Waste Disposal (Revision 1, 03/01/08)	NA	Waste Disposal	NA/ Disposal	Microbial Insights	N
KBSOP01VOC	Analytical Standard Operating Procedure No. 1, Determination of VOCs by Purge and Trap Gas Chromatography/ Mass Spectrometry Method 8260B (Revision 4, July 2008)	Screening	Groundwater and sediment pore water/VOCs	GC/MS	KB Labs	N

Note:  
Copies of all the Laboratory SOPs listed in this table are included in Appendix B.

**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
GC/MS VOCs	Initial calibration (ICAL) – a minimum of a 5-point calibration is prepared for all target analytes.	Calibrate the instrument when it is received and after a major change 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 $\geq 0.30$ . The average RFs for the SPCCs: chloromethane, 1,1-Dichloroethane and bromoform must be $\geq 0.10$ ;  The Percent Relative Standard Deviations (%RSDs) for RFs of Calibration Check Compound (CCCs) must be $\leq 30\%$ ; and the %RSDs must be $< 15\%$ for all compounds. If not met:  Option 1) Linear least squares regression: Linear Regression Correlation Coefficient (r) must be $\geq 0.995$ .  Option 2) Non-linear regression: coefficient of determination ( $r^2$ ) must be $\geq 0.99$ (6 points for second order).	Correct problem and repeat ICAL. Do not analyze samples until ICAL passes criteria.	Analyst, Department Manager	Empirical SOP-202
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL prior to beginning a sample run.	Percent Recovery (%R) of each analyte must be within 80-120% of true value.	Correct problem and verify ICV. Reanalyze initial calibration. Do not analyze samples until ICV passes criteria.	Analyst, Department Manager	
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune and before sample analysis.	All target compounds must be $< 20$ Percent Difference or Percent Drift (%D). Average RFs for SPCCs must be $> 0.10$ & $0.30$ (compounds as listed above in initial calibration block).	Repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	Tune Verification - BFB	Prior to ICAL and at the beginning of each 12 hour analytical sequence.	Criteria listed in SOP-202. Must meet the ion abundance criteria required by the method.	Retune and/or clean source. No samples may be accepted without a valid tune.	Analyst, Department Manager	
GC/MS SVOCs and PAHs	ICAL – a minimum of a 5-point calibration is prepared for all target analytes.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	Scan :Average RF must be $\geq 0.050$ ; %RSD must be $< 15\%$ for all compounds. 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-201
	ICV – Second Source	Once after each initial calibration prior to beginning a sample run.	%R of each analyte must be within 80-120% of true value.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluoro-triphenyl-phosphine (DFTPP) tune.	Scan: %D for all target compounds must be $\leq 20\%$ ; Average RF for must be $\geq 0.050$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Tune Verification – DFTPP	At the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method.	Retune and/or clean source.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
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 %RSD for each analyte 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.	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	
GC-FID Dissolved Gases	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 as needed.	$r$ must be $\geq 0.995$ or $r^2$ 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 - 236
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R of all analytes must be within 75-125% of true value.	Correct problem and verify second source standard. Reanalyze ICAL.	Analyst, Department Manager	

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency of Calibration</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Person Responsible for CA</b>	<b>SOP Reference</b>
GC-FID Dissolved Gases	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R of all analytes must be within 80-120% of true value.	Repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	
IC Anions	ICAL – A minimum of a 3-point calibration is prepared and a linear range is established for all target analyses.	Perform after major instrument maintenance and upon failure of second consecutive CCV.	The %RSD must be < 15% over linear range, or r must be $\geq 0.995$ .	Correct the problem, then repeat ICAL.	Analyst, Department Manager	Empirical SOP-145
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value and retention times (RTs) must be within appropriate windows.	Correct problem and verify ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value and all project analytes must be within established RT windows.	Correct problem and verify CCV. If that fails, repeat ICAL and reanalyze all samples since the last successful CCV.	Analyst, Department Manager	
TOC Analyzer TOC	ICAL	Each analytical sequence.	r must be $\geq 0.995$ .	Correct the problem, then repeat ICAL.	Analyst, Department Manager	Empirical SOP-221
	ICV – Second Source	Each analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
	CCV	Every 10 samples and at the end of the analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency of Calibration</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Person Responsible for CA</b>	<b>SOP Reference</b>
ICP- AES TAL Metals and Molybdenum, Total and Dissolved Iron and Manganese, and Lead	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 $\geq 0.995$ .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP-100/105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 90-110% of the true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration.	Analyst, Department Manager	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst, Department Manager	
	Low-Level Check Standard	Daily after ICAL and before samples.	The %R must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning and end of an analytical run and after each batch of 20 samples.	ICS A recoveries must be within the absolute value of the LOD; and ICS B %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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Mercury Analyzer (CVAA)	Initial Calibration (minimum of 5-point calibration)	Upon instrument receipt, major instrument change, and at the start of each day prior to sample analysis.	r must be $\geq 0.995$ or RSD for RFs must be $\leq 20\%$ .	Correct problem and repeat ICAL. Do not analyze samples until ICAL passes criteria.	Analyst, Department Manager	Empirical SOP-103/104
	ICV (Second Source)	Following ICAL, before beginning a sample analysis.	%Rs must be within 90-110% of true values.	Correct problem and verify ICV. If that fails, correct problem, repeat ICAL and reanalyze all samples analyzed since last successful CCV.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample analysis sequence.	No analytes detected > LOD.	Correct problem and repeat calibration.	Analyst, Department Manager	
	CCV	Every 10 samples and at end of analytical	%Rs must be within 80-120% of true values.	Correct problem and rerun ICV. If that fails, correct problem, repeat ICAL and reanalyze all samples analyzed since last successful CCV.	Analyst, Department Manager	
Buret Dissolved Sulfide	Standardization	Daily prior to sample analysis.	Standardized using 0.25 N sodium thiosulfate.	An acceptable titrant is compared against an independent source identified as an LCS/ICV.	Analyst, Department Manager	Empirical SOP-153
	ICV – Second Source	After ICAL and each analytical sequence.	The %R must be within 80-120% of the true value.	Recalibrate.	Analyst, Department Manager	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
pH Meter Alkalinity	Standardization	Daily prior to sample analysis.	Standardize using pH 7 and pH 4, adjust as needed, and reread. Must be within $\pm 0.05$ pH units to proceed.	Restandardize.	Analyst, Department Manager	Empirical SOP-154
	Buffer check	Check every 3 hours.	Must be within $\pm 0.20$ pH units.	Restandardize and rerun samples.	Analyst, Department Manager	
GC/MS VOC Screening by Mobile Lab	ICAL – a minimum of a 5-point calibration is prepared for all target analytes.	Prior to any sample analysis.	The %RSD of target analyte RFs must be $\leq 15\%$ . Minimum mean RFs of SPCCs as listed in SW-846 8260B must be met during the ICAL. The %RSDs of CCC RFs during ICAL must be $\leq 30\%$ .	Correct problem and repeat ICAL. Do not analyze samples until ICAL passes criteria.	Analyst	KB Labs SOP01VOC
GC/MS VOC Screening by Mobile Lab	CCV -a midlevel standard run every 12 hours prepared from separate source from calibration standards.	Daily before sample analysis and every 12 hours of analysis time.	RF criteria for SPCCs the same as during ICAL. RF of CCCs must be $<20\%$ from ICAL.	Rerun CCV. Then rerun ICAL, if necessary.	Analyst	KB Labs SOP01VOC
	BFB Tune	Prior to ICAL and at the beginning of each 12 hour analytical sequence.	Criteria listed in SW-846 8260B.	Retune and/or clean source.	Analyst	

Notes:

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

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

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in 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 and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-202
GC/MS	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	SVOCs and PAHs	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-201
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals and Molybdenum, Total and Dissolved Iron, and Lead	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-100/105
CVAA	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 Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-103/104
GC/FID	Replace Septa, Check gases, Clean FID, replace TCD filaments, Change activated carbon, Bake out column.	Dissolved Gases	Visual inspection of septa, FID, Filaments	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst	Empirical SOP-236

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
TOC Analyzer	Replace sample tubing, clean sample boat, replace syringe.	TOC	Tubing, sample boat, syringe	As needed.	Must meet ICAL and CCV criteria.	Repeat maintenance activity of remove from service.	Analyst, Laboratory Area Supervisor	Empirical SOP-221
IC Dionex	Check and clean segments weekly, clean reagent tubes monthly. Change syringes, eluent, and dispensing needle, all as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Anions	Check tubing. Verify that Chromatography is acceptable.	Prior to initial calibration or as necessary.	Must meet ICAL and CCV criteria.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-145
Buret	Check Buret for any cracks or chips. Rinse buret prior to each use and at the end of each day.	Dissolved Sulfide	Visual inspection for cracks or chips.	Each use.	NA.	Remove from service.	Analyst, Department Manager	Empirical SOP-153
GC/FID	Check pressure and gas supply daily. Bake out column, change septa, liner, seal as needed, cut column as needed.	TRPH (FL-PRO)	Liner, seal, septum, column	As needed.	The compounds must be < 25%D.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration.	Analyst, Laboratory Area Supervisor	Empirical SOP-338

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

<b>Sample Collection, Packaging, and Shipment</b>
Sample Collection (Personnel/Organization): FOL or designee/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/ Tetra Tech
Type of Shipment/Carrier: Federal Express
<b>Sample Receipt and Analysis</b>
Sample Receipt (Personnel/Organization): Sample Custodians/ Empirical, KB Labs, and Microbial Insights
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical, KB Labs, and Microbial Insights
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/ Empirical and Microbial Insights; KB Labs
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab/ Empirical and Microbial Insights; KB Labs
<b>Sample Archiving</b>
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
<b>Sample Disposal</b>
Personnel/Organization: Sample Custodians/ Empirical and Microbial Insights

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

### **27.1 FIELD SAMPLE CUSTODY PROCEDURES**

Sample Chain-of-Custody forms will be completed per Tetra Tech SOP SA-6.3. An example is included in Appendix A.

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.

### **27.2 SAMPLE NOMENCLATURE**

Worksheet #18 presents the sample nomenclature for the field and lists QA/QC samples to be collected.

### **27.3 SAMPLE COLLECTION AND DOCUMENTATION**

Documentation of field observations will be recorded in a field logbook and/or on field log sheets including sample collection logs and boring logs. Bound, water-resistant field logbooks will be used for this project. Logbook pages will be numbered sequentially, and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details. Other observations and activities will be recorded in the field logbook. Daily instrument calibration will be recorded in instrument calibration logs.

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

- Site name and location
- Date and time
- Personnel and their affiliations
- Weather conditions
- Activities associated with sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches monitoring well layout, if different than sampling plan figures
- Visitor names, affiliations, and arrival and departure times
- Health and safety issues including PPE

## **27.4 SAMPLE PACKAGING AND SHIPPING**

Samples will be prepared for shipping using the following guidelines:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (i.e., Ziploc-type bag), and seal bag.
- Place sample in a sturdy cooler that has been lined with a large plastic bag (i.e., garbage bag). Drain plugs on coolers should be taped shut.
- Place a temperature check indicator provided by the laboratory in each cooler to be shipped.
- Pack with sufficient cushioning materials, such as bubble wrap, to minimize the possibility of the container breaking.
- If cooling is required, pack sample containers in ice to adequately cool sample to 4°C.
- Seal large liner bag by taping or knotting open end.
- Tape the original top, signed copy of the chain-of-custody form shall be placed in a large Ziploc-type bag inside the lid of the shipping cooler. If multiple coolers are sent but samples are included on one chain-of-custody form, the chain-of-custody form should then indicate how many coolers are included with the shipment.
- Close and seal the outside of shipping cooler using strapping tape. Place custody seals across the lid and body of cooler and under strapping tape to prevent tampering while in transit. No Department of Transportation (DOT) marking is required.

## **27.5 SAMPLE HANDLING AND TRACKING SYSTEM**

Sample handling is described in Worksheet #26. Samples must be delivered to the laboratory via a public courier (e.g., Federal Express). Samples must be sent to the laboratory within 24 hours of being collected. Under no circumstances should sample holding times be exceeded.

## **27.6 SAMPLE CUSTODY**

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate written record that traces the possession and handling of the sample. This documentation is referred to as the chain-of-custody form. The chain of custody begins at the time of sample collection. The laboratory will provide forms that will be used for chain-of-custody documentation.

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. Each page of the form is signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample will each retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the analytical 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 shipping courier. When transferring samples, the individuals relinquishing and receiving the samples will each sign the chain-of-custody form. The date and time will be recorded to each time the samples change hands. Once delivered to the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix B.

#### **27.6.1 Field Sampling Custody Requirements**

Field Sample Custody Procedures (sample collections, packaging, and shipping to laboratory) will be conducted according to Tetra Tech SOP SA-6.3 (Appendix A). Following sample collections in 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 to protect the bottle ware during shipment and to prevent cross contamination should a bottle break in transit. The cooler will be secured using duct tape or clear packaging tape along with two signed custody seals. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis.

The Tetra Tech FOL is responsible for the care and custody of the samples 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 form documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier).

## **27.6.2**    **Laboratory**

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical, KB Labs, and Microbial Insights 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)

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No analytes > ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination. Rerun method blank prior to analysis of samples if possible. Evaluate the samples and associated QC: if blank results are above LOQ, then report sample results which are < LOQ or > 10X the blank concentration. Reanalyze blank and samples >LOQ and < 10X the blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	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, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (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 ≤ 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, Laboratory Department Manager, and Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP 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
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) (not required)	One is performed for each batch of up to 20 samples.	DOD QSM limits are used. If LCSD performed - The RPD between LCS and LCSD must be $\leq 30\%$ .	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	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 ICAL standard for each IS.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No target compounds > ½ the LOQ, except common lab contaminants, which must be < the LOQ.	(1) Investigate source of contamination (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	Six per sample: 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14 For Low PAHs, 2-Fluorobiphenyl and Terphenyl-d14 only	%Rs must meet the DOD QSM version 4.1 limits as per Appendix G of the DoD QSM.  <i>For Low PAH – water limits are 34-167% and soil limits are 14-129%.</i>	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, then check instrument performance; if problem is found, then correct and reanalyze. (3) If still out, then re-extract and analyze sample. (4) If reanalysis is out, then flag data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples of similar matrix.	%Rs should meet the DOD QSM version 4.1 limits as per Appendix G of the DoD QSM, except Low PAHs which are provided in Appendix B.  RPD between MS and MSD should be ≤ 30%.	Corrective Action will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias / Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the DOD QSM version 4.1 limits as per Appendix G of the DoD QSM, except Low PAHs which are provided in Appendix B.  If LCSD performed - The RPD between LCS and LCSD must be $\leq 30\%$ .	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable, then narrate. If the LCS recoveries are high but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias/ Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
ISs	Six per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	RTs for ISs must be within $\pm 30$ seconds and the response areas must be within -50% to +100% of the midpoint ICAL standard for each IS.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

**SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)**

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.	No analytes detected > ½ the LOQ, except common lab contaminants, which must be < LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value; then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples.	The %R must be within 80-120%.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS	One per preparation batch of 20 or fewer samples of similar matrix.	The %R should be within 80-120%.	Flag results for affected analytes for all associated samples with "N".	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Duplicate Sample	One per preparation batch of 20 or fewer samples of similar matrix.	The RPD should be within ≤20%.	Narrate any results that are outside control limits.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

**SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)**

<b>Matrix</b>	<b>Groundwater, Soil, and Aqueous QC Blanks</b>					
<b>Analytical Group</b>	<b>TAL Metals (Including Mercury) and Molybdenum, Total and Dissolved Iron, and Lead</b>					
<b>Analytical Method/ SOP Reference</b>	<b>SW-846 6010C/7470A/7471A Empirical SOP-103, 104, 105</b>					
<b>QC Sample</b>	<b>Frequency/ Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
Serial Dilution	One is performed for each preparation batch with sample concentration(s) > 50x LOQ.	The result must agree within $\pm 10\%$ of the original sample result.	Perform Post Digestion Spike.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or analyte concentration(s) in all samples < 50x LOD.	%R must be within 75-125% of the true value.	Flag results of samples of same matrix as estimates in SDG narrative.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

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 batch of up to 20 samples.	Analyte concentration must be <½ LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	%R must be within 80-120% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples.	%R should be within 75-125% of the expected value. RPD should be ≤ 30%.	Contact client for guidance.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

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 batch of up to 20 samples.	Analyte concentration must be <½ LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	%R must be within 80-120% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples.	%R should be within 75-125% of the expected value. RPD should be ≤ 20%.	Contact client for guidance.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

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 batch of up to 20 samples.	Analyte concentration must be <math>\leq \frac{1}{2}</math> LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	%R must be within 90-110% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples.	%R should be within 80-120% of the expected value. RPD should be $\leq 20\%$ .	Contact client for guidance.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

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 batch of up to 20 samples.	Analyte concentration must be <math>\leq \frac{1}{2}</math> LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	%R must be within 80-120% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples.	%R should be within 75-125% of the expected value. RPD should be $\leq 20\%$ .	Contact client for guidance.	Analyst, Laboratory Area Supervisor, and Data Validator	Accuracy/ Bias Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

Matrix	Soil and Aqueous QC Blanks					
Analytical Group	TRPH					
Analytical Method/ SOP Reference	FL-PRO/ Empirical <a href="#">SOP-338</a>					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Must be <1/2 the LOQ.	Re-clean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	2 per sample: 2-Fluorobiphenyl o-Terphenyl	%Rs must pass the established laboratory criteria.	(1) Prepare again and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy /Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of 20 or less	%Rs must meet limits of 55-118% for water and 50-140% for 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, Laboratory Supervisor and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples.	%Rs should meet the limits of 55-118% for water and 50-140% for water.  The RPD between MS and MSD should be ≤ 30%.	(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, Laboratory Supervisor and Data Validator	Precision / Accuracy /Bias	Same as Method/SOP QC Acceptance Limits.

**SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)**

Matrix	Groundwater					
Analytical Group	Alkalinity					
Analytical Method/SOP Reference	SM2320B Empirical SOP-154					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Analyte concentration must be $< \frac{1}{2}$ LOQ.	Correct problem, re-prepare, and reanalyze along with all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination/ Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	%R must be within 80-120% of true value.	Correct problem, re-prepare, and reanalyze the LCS along with all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples.	%R should be within 75-125% of the expected value.  RPD should be $\leq 20\%$ .	Contact client for guidance.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

**Please note:** Dehalococoides and reductase genes information is not relevant to this worksheet.

SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)

<b>Matrix</b>	Groundwater, Storm Sewer Water, Sediment Pore Water, Surface Water, and Aqueous QC Blanks					
<b>Analytical Group</b>	VOCs Screening Mobile Lab					
<b>Analytical Method/ SOP Reference</b>	SW-846 8260B KBSOP01VOC					
<b>QC Sample</b>	<b>Frequency/ Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
Method Blank	One per daily analysis batch.	No analytes > 1/2 of the LOQ.	Bake out purge trap system, change adsorbent trap. Reprepare and reanalyze method blank and associated samples.	Analyst	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: 1,4-dichlorobenzene 1,2-dichloroethane-d4 Toluene-d8 BFB	Should be within limits established by lab or method.	Reanalyze sample. If one or more still remain outside criteria, then recalibrate and or remake surrogate solution.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per 20 samples of similar matrix.	Should be within limits established by lab.	Check LCS to see if matrix effects apply.	Analyst	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

**SAP Worksheet No. 28 – Laboratory QC Samples Table (Continued)**

<b>Matrix</b>	Groundwater, Storm Sewer Water, Sediment Pore Water, Surface Water, and Aqueous QC Blanks					
<b>Analytical Group</b>	VOCs Screening Mobile Lab					
<b>Analytical Method/ SOP Reference</b>	SW-846 8260B KBSOP01VOC					
<b>QC Sample</b>	<b>Frequency/ Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
LCS	One per daily analysis batch.	Must be within limits established by lab.	Reprepare and reanalyze LCS. Reanalyze associated samples.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs for ISs must be within ± 30 seconds and the response areas must be within -50% to +100% of the last calibration check.	Reanalyze sample. If one or more still remain outside criteria, recalibrate.	Analyst	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

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

Document	Where Maintained
<p><b><u>Field Documents</u></b></p> <ul style="list-style-type: none"> <li>• Field Logbook</li> <li>• Field Sample Forms</li> <li>• Chain of Custody Records</li> <li>• Air bills</li> <li>• Sampling Instrument Calibration Logs</li> <li>• Sampling Notes</li> <li>• Photographs</li> <li>• Field Task Modification Forms</li> <li>• This SAP</li> <li>• HASP</li> </ul>	<p>Field documents will be maintained in the project file located in the Tetra Tech Jacksonville office.</p>
<p><b><u>Laboratory Documents and Records in the form of analytical data package:</u></b></p> <ul style="list-style-type: none"> <li>• Sample receipt/login form</li> <li>• Sample storage records</li> <li>• Sample preparation logs</li> <li>• Equipment calibration logs</li> <li>• Sample analysis run logs</li> <li>• Reported results for standards, QC checks, and QC samples</li> <li>• Data completeness checklists</li> <li>• Telephone logs</li> <li>• Extraction/clean-up records</li> <li>• Raw data</li> </ul>	<p>Laboratory documents will be included in the hardcopy and portable documents format (pdf) deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Pittsburgh project file and in long-term data package storage at a third-party professional document storage firm.</p> <p>Electronic data results will be maintained in a database on a password protected SQL server.</p>
<p><b><u>Assessment Findings</u></b>            Field Sampling Audit Checklist (if conducted)            Analytical Audit Checklist (if conducted)            Data Validation Memoranda (includes tabulated data summary forms)</p>	<p>All assessment documents will be maintained in the Pittsburgh Tetra Tech office.</p>
<p><b><u>Reports</u></b>            RI/FS Addendum Report</p>	<p>All reports the will be stored in hardcopy in the Tetra Tech Jacksonville project file and electronically in the server library.</p>

**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)
Groundwater and Aqueous QC Blanks	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Kim Kostzer Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	NA
	SVOCs and PAHs	See Worksheet #18	SW-846 8270D			
	TAL Metals plus Molybdenum, Total and Dissolved Iron, and Lead	See Worksheet #18	SW-846 6010C/7470A			
	MNA Parameters					
	Alkalinity	See Worksheet #18	SM 2320B			
	Dissolved Sulfide	See Worksheet #18	SM 4500S F			
	TOC	See Worksheet #18	SM 5310C			
	Anions	See Worksheet #18	EPA 300.0			
	Dissolved Gases	See Worksheet #18	RSK SOP 175			
Surface Soil	VOCs	See Worksheet #18	SW-846 8260B			
	TRPH	See Worksheet #18	FDEP FL-PRO			
	SVOCs and PAHs	See Worksheet #18	SW-846 8270D			
	TAL Metals plus Molybdenum, Total and Dissolved Iron, and Lead	See Worksheet #18	SW-846 6010C/7471A			

<b>Matrix</b>	<b>Analytical Group</b>	<b>Sample Locations/ID Numbers</b>	<b>Analytical Method</b>	<b>Data Package Turnaround Time</b>	<b>Laboratory / Organization</b> (name and address, contact person and telephone number)	<b>Backup Laboratory / Organization</b> (name and address, contact person and telephone number)
Groundwater	Dehalococcoides and reductase genes	See Worksheet #18	Laboratory Proprietary	21 calendar days	Anita Biernacki Microbial Insights 2340 Stock Creek Boulevard Rockford, TN 37853-3044 (865) 573-8188, x108	NA
Groundwater, storm sewer water, sediment pore water, and surface water	VOCs – mobile laboratory screening	See Worksheet #18	SW-846 8260B	Results in 24 hours	Todd Romero <a href="mailto:toddr@kbmobilelabs.com">toddr@kbmobilelabs.com</a> KB Labs, Inc. 25132 SW 1st Ave Newberry, Florida 32669 Telephone (352) 472-5830 Fax (352) 472-5832	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 <sup>1</sup>	2 years	External	DoD ELAP	DoD ELAP Accrediting Body	Laboratory QA Manager or Laboratory Manager	Laboratory QAM or Laboratory Manager	Laboratory QAM or Laboratory Manager

Notes:

<sup>1</sup> Empirical Laboratories is DoD ELAP accredited. A copy of Empirical's accreditation is included in Appendix B. Microbial Insights is specialty support laboratory and does not require DoD ELAP accreditation. KB Labs, Inc. (mobile laboratory) is NELAP accredited in the state of Florida.

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

<b>Assessment Type</b>	<b>Nature of Deficiencies Documentation</b>	<b>Individual(s) Notified of Findings</b> (name, title, organization)	<b>Timeframe of Notification</b>	<b>Nature of CA Response Documentation</b>	<b>Individual(s) Receiving CA Response</b> (name, title, organization)	<b>Timeframe for Response</b>
Laboratory System Audit	Written audit report	Rick Davis, Laboratory Manager, Empirical Randy Ward, Laboratory QAM, Empirical	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

**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 3 weeks of receipt of laboratory data package	DVM and Staff Chemists, Tetra Tech	PM and project file, Tetra Tech
Major analysis problem identification (internal memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem (on the same day)	CLEAN QAM, Tetra Tech	PM, CLEAN QAM, Program Manager, 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
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical, Microbial Insights, and KB Labs	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.	Internal	Sampler and FOL, Tetra Tech
	Each Laboratory's 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, KB Labs, and Microbial Insights 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 MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Analytical SOPs/ Analytical Data Packages	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, Empirical and Microbial Insights Data specialist, KB Labs

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	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 verbally or via e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical and Microbial Insights Data specialist, KB Labs
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
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
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 and Microbial Insights Data specialist, KB Labs
	Each data package will be verified for completeness by the Tetra Tech Data Validator. Missing information will be requested by the Tetra Tech Data Validator from the Laboratory PM.	External	Data Validators, Tetra Tech

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/ Sample Log Sheets	Sample Coordinates - 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	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	<p>Accuracy - Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.</p> <p>Precision - Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.</p> <p>Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.</p> <p>Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final SQL database.</p>	Project Chemist or Data Validators, Tetra Tech

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

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

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Aqueous	VOCs, Dissolved Gases, SVOCs, PAHs, TRPH	100% Limited data validation will be performed. SW-846 8260B, RSK SOP 175, 8270D, 8270D SIM, and FL-PRO method specific criteria and those criteria listed in Worksheets #12, #15, #24, and #28. If not included in Worksheets #12, #15, #24, or #28, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Aqueous	TAL Metals plus Molybdenum, Total and Dissolved Iron, and Lead	100% Limited* data validation will be performed. SW-846 6010B method specific criteria and those listed in Worksheets #12, #15, #24, and #28. If not included in Worksheets #12, #15, #24 or #28, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA 540-R-04-004, October 2004 will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater, surface water, soil, and sediment	Anions, Alkalinity, TOC, Dissolved Sulfide	Method-specific criteria listed in Worksheets #12, #15, #24, and #28.	Data Validation Specialist, Tetra Tech

\* Limited data validation. Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding times, Calibrations, Blank Contamination, & 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 data validation report is prepared.

Dehalococoides and VOC mobile laboratory data reports will not be validated.

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

**Data Usability Assessment**

The usability of the data directly affects whether project objectives can be achieved. At a minimum the following characteristics will be evaluated. 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 Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM and Project 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 Project Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

**Precision**

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

**Accuracy**

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

**Representativeness**

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

## Data Usability Assessment

### Comparability

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

### Sensitivity

The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Tetra Tech 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. Quantitative assumptions include assumptions related to data distributions (e.g., normal versus log-normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated data indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

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

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members identified by the Tetra Tech PM will assess whether the data collectively support the attainment of project objectives. 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

### Data Usability Assessment

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, the USEPA RPM, and the FDEP RPM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

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

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

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**APPENDIX A**

**FIELD STANDARD OPERATING PROCEDURES AND  
FIELD DATA SHEETS**



# CONTAINER SAMPLE & INSPECTION SHEET

Project Site Name: _____	Sample ID No. _____
Project Number: _____	Sampled By: _____
Site Identification: _____	C.O.C. No.: _____
Container Number(s): _____	Concentration: <input type="checkbox"/> High
Sample Type: <input type="checkbox"/> Grab	<input type="checkbox"/> Medium
<input type="checkbox"/> Composite	<input type="checkbox"/> Low

CONTAINER SOURCE	CONTAINER DESCRIPTION
------------------	-----------------------

**DRUM:**

Bung Top

Lever Lock

Bolted Ring

Other \_\_\_\_\_

COLOR: \_\_\_\_\_

CONDITION: \_\_\_\_\_

**TANK:**

Plastic

Metal

Other \_\_\_\_\_

MARKINGS: \_\_\_\_\_

VOL. OF CONTENTS: \_\_\_\_\_

OTHER: \_\_\_\_\_

OTHER: \_\_\_\_\_

CONTAINER DISPOSITION	CONTENTS DESCRIPTION
-----------------------	----------------------

**SAMPLED:** \_\_\_\_\_

**OPENED BUT NOT SAMPLED:**  
Reason \_\_\_\_\_

**NOT OPENED:**  
Reason \_\_\_\_\_

**SINGLE PHASED:** \_\_\_\_\_

**MULTIPHASE :**

	<b>Layer 1</b>	<b>Layer 2</b>	<b>Layer 3</b>
Phase (Sol. or Liq.)	_____	_____	_____
Color	_____	_____	_____
Viscosity	L, M or H	L, M or H	L, M or H
% of Total Volume	_____	_____	_____

MONITOR READING:	SAMPLE and /or INSPECTION DATE & TIME:
------------------	--

\_\_\_\_\_

\_\_\_\_\_ HRS.

**METHOD:** \_\_\_\_\_

SAMPLER(S) and / or INSPECTOR(S) SIGNATURE:	ANALYSIS:
---	-----------

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_







# Tetra Tech NUS, Inc.

PROJECT: \_\_\_\_\_ LOCATION: \_\_\_\_\_  
 JOB & CTO #: \_\_\_\_\_ MOBILIZATION DATE: \_\_\_\_\_  
 PROJECT MANAGER: \_\_\_\_\_ RETURN DATE: \_\_\_\_\_

<b>FIELD PROJECT PRE-MOBILIZATION CHECKLIST</b>	
<b>TRAVEL</b>	<b>MISCELLANEOUS</b>
<input type="checkbox"/> Airline reservations <input type="checkbox"/> Hotel reservations/BOQs <input type="checkbox"/> Vehicle rental <input type="checkbox"/> Itinerary <input type="checkbox"/> Phone/pager number	<b>Schedule</b> <input type="checkbox"/> Plan field operations w/ Project manager <b>Documents for Field Program</b> <input type="checkbox"/> Logbook(s) <input type="checkbox"/> Field Sampling plan <input type="checkbox"/> Health & Safety plan <input type="checkbox"/> Maps <input type="checkbox"/> H & S Guidance Manual <b>Authorization</b> <input type="checkbox"/> Kick-off meeting held <input type="checkbox"/> Gov't rate letter <input type="checkbox"/> H&S/OSHA 40-hour certificate <input type="checkbox"/> 8-Hour Refresher Training Certificate <input type="checkbox"/> Medical Clearance Letter <input type="checkbox"/> Supervisory Training Certificate <input type="checkbox"/> Health & Safety Clearance Letter <input type="checkbox"/> Full-size OSHA Poster
<b>DRILLING/DPT/SURVEY</b>	<b>HYDROGEOLOGY EQUIPMENT</b>
<b>Subcontractor</b> <input type="checkbox"/> POC phone #/address <input type="checkbox"/> Drill Specification RFP <input type="checkbox"/> Contact (time & place to meet) <input type="checkbox"/> Confirm subcontract w/ TtNUS Procurement <input type="checkbox"/> Health and Safety documentation for all personnel on site <input type="checkbox"/> Copy of Drillers license <input type="checkbox"/> Well / boring permits  <b>Utilities (2 weeks lead time)</b> <input type="checkbox"/> Contact Site POC (Date: _____) <input type="checkbox"/> Contact Local "Call Before You Dig" <input type="checkbox"/> Utility Clearance Form <b>Forms</b> <input type="checkbox"/> Boring logs / Test Pit logs <input type="checkbox"/> Well construction / development forms <input type="checkbox"/> Daily activity forms <input type="checkbox"/> IDW inventory <input type="checkbox"/> IDW drum labels <input type="checkbox"/> Chemical Inventory <input type="checkbox"/> MSDS's	<input type="checkbox"/> Slug test/pumping test forms <input type="checkbox"/> Groundwater elevation data sheets <input type="checkbox"/> Graph paper <input type="checkbox"/> Data Logger/transducer/data cable <input type="checkbox"/> Existing well construction & water level data <input type="checkbox"/> M-Scope, slug
<b>EQUIPMENT MOBILIZATION</b>	<b>SHIPPING</b>
<input type="checkbox"/> Equipment Requisition form completed / equipment ordered <input type="checkbox"/> 3rd Party rental / misc. equipment ordered <input type="checkbox"/> Equipment calibration forms <input type="checkbox"/> Span / calibration gas and regulator	<b>Forms</b> <input type="checkbox"/> FedEx Airbills, local dropoff location & hours <input type="checkbox"/> FedEx Gov. Acct# (1771-8058-0) <input type="checkbox"/> Lab Shipping Labels <input type="checkbox"/> Warehouse Shipping Labels <input type="checkbox"/> Blank Labels  <b>Supplies</b> <input type="checkbox"/> Tape <input type="checkbox"/> Packing materials <input type="checkbox"/> Baggies, Large garbage bags
<b>SAMPLING</b>	<b>OTHER</b>
<b>Forms</b> <input type="checkbox"/> Sample log sheets <input type="checkbox"/> Low-flow purge data sheets <input type="checkbox"/> COC records <input type="checkbox"/> COC seals <input type="checkbox"/> Sample labels (from database group) <b>Laboratory</b> <input type="checkbox"/> POC address/phone# <input type="checkbox"/> Order bottles / preservatives <input type="checkbox"/> Shipping address, also check Sat. address <input type="checkbox"/> Bottle & preservation req'ts from lab	<input type="checkbox"/> Site POC name/phone # <input type="checkbox"/> Personnel information to POC <input type="checkbox"/> Mobilization schedule to POC <input type="checkbox"/> Site access authorizations <input type="checkbox"/> Field office / trailer arrangements made <input type="checkbox"/> Electric, phone hookups arranged <input type="checkbox"/> Steel-toed boots, safety glasses, & hard hat <input type="checkbox"/> First aid equipment <input type="checkbox"/> Insect repellent  <input type="checkbox"/> _____ <input type="checkbox"/> _____

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.















**TETRA TECH NUS**  
**FIELD TASK MODIFICATION REQUEST FORM**

Project/Installation Name \_\_\_\_\_ CTO & Project Number \_\_\_\_\_ Task Mod. Number \_\_\_\_\_

Modification To (e.g. Work Plan) \_\_\_\_\_ Site/Sample Location \_\_\_\_\_ Date \_\_\_\_\_

Activity Description: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Reason for Change: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Recommended Disposition: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Field Operations Leader (Signature) \_\_\_\_\_ Date \_\_\_\_\_

Approved Disposition: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Project/Task Order Manager (Signature) \_\_\_\_\_ Date \_\_\_\_\_

Distribution:

Program/Project File – \_\_\_\_\_  
Project/Task Order Manager – \_\_\_\_\_  
Field Operations Leader – \_\_\_\_\_

Other: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_



# Tetra Tech NUS, Inc.

PROJECT: \_\_\_\_\_ LOCATION: \_\_\_\_\_  
 JOB & CTO #: \_\_\_\_\_ MOBILIZATION DATE: \_\_\_\_\_  
 PROJECT MANAGER: \_\_\_\_\_ RETURN DATE: \_\_\_\_\_

<b>FIELD PROJECT DEMOBILIZATION CHECKLIST</b>	
<b>TRAVEL</b>	<b>MISCELLANEOUS</b>
<input type="checkbox"/> Airline reservations <input type="checkbox"/> Hotel reservations/BOQs <input type="checkbox"/> Vehicle rental <input type="checkbox"/> Itinerary <input type="checkbox"/> Phone/pager number	<b>Schedule</b> <input type="checkbox"/> Plan field operations w/ Project manager <b>Documents for Field Program</b> <input type="checkbox"/> Logbook(s) <input type="checkbox"/> Field Sampling plan <input type="checkbox"/> Health & Safety plan <input type="checkbox"/> Maps <input type="checkbox"/> H & S Guidance Manual <b>Authorization</b> <input type="checkbox"/> Kick-off meeting held <input type="checkbox"/> Gov't rate letter <input type="checkbox"/> H&S/OSHA 40-hour certificate <input type="checkbox"/> 8-Hour Refresher Training Certificate <input type="checkbox"/> Medical Clearance Letter <input type="checkbox"/> Supervisory Training Certificate <input type="checkbox"/> Health & Safety Clearance Letter <input type="checkbox"/> Full-size OSHA Poster
<b>DRILLING/DPT/SURVEY</b>	<b>HYDROGEOLOGY EQUIPMENT</b>
<b>Subcontractor</b> <input type="checkbox"/> POC phone #/address <input type="checkbox"/> Drill Specification RFP <input type="checkbox"/> Contact (time & place to meet) <input type="checkbox"/> Confirm subcontract w/ TtNUS Procurement <input type="checkbox"/> Health and Safety documentation for all personnel on site <input type="checkbox"/> Copy of Drillers license <input type="checkbox"/> Well / boring permits  <b>Utilities (2 weeks lead time)</b> <input type="checkbox"/> Contact Site POC (Date: _____) <input type="checkbox"/> Contact Local "Call Before You Dig" <input type="checkbox"/> Utility Clearance Form <b>Forms</b> <input type="checkbox"/> Boring logs / Test Pit logs <input type="checkbox"/> Well construction / development forms <input type="checkbox"/> Daily activity forms <input type="checkbox"/> IDW inventory <input type="checkbox"/> IDW drum labels <input type="checkbox"/> Chemical Inventory <input type="checkbox"/> MSDS's	<input type="checkbox"/> Slug test/pumping test forms <input type="checkbox"/> Groundwater elevation data sheets <input type="checkbox"/> Graph paper <input type="checkbox"/> Data Logger/transducer/data cable <input type="checkbox"/> Existing well construction & water level data <input type="checkbox"/> M-Scope, slug
<b>EQUIPMENT MOBILIZATION</b>	<b>SHIPPING</b>
<input type="checkbox"/> Equipment Requisition form completed / equipment ordered <input type="checkbox"/> 3rd Party rental / misc. equipment ordered <input type="checkbox"/> Equipment calibration forms <input type="checkbox"/> Span / calibration gas and regulator	<b>Forms</b> <input type="checkbox"/> FedEx Airbills, local dropoff location & hours <input type="checkbox"/> FedEx Gov. Acct# (1771-8058-0) <input type="checkbox"/> Lab Shipping Labels <input type="checkbox"/> Warehouse Shipping Labels <input type="checkbox"/> Blank Labels  <b>Supplies</b> <input type="checkbox"/> Tape <input type="checkbox"/> Packing materials <input type="checkbox"/> Baggies, Large garbage bags
<b>SAMPLING</b>	<b>OTHER</b>
<b>Forms</b> <input type="checkbox"/> Sample log sheets <input type="checkbox"/> Low-flow purge data sheets <input type="checkbox"/> COC records <input type="checkbox"/> COC seals <input type="checkbox"/> Sample labels (from database group) <b>Laboratory</b> <input type="checkbox"/> POC address/phone# <input type="checkbox"/> Order bottles / preservatives <input type="checkbox"/> Shipping address, also check Sat. address <input type="checkbox"/> Bottle & preservation req'ts from lab	<input type="checkbox"/> Site POC name/phone # <input type="checkbox"/> Personnel information to POC <input type="checkbox"/> Mobilization schedule to POC <input type="checkbox"/> Site access authorizations <input type="checkbox"/> Field office / trailer arrangements made <input type="checkbox"/> Electric, phone hookups arranged <input type="checkbox"/> Steel-toed boots, safety glasses, & hard hat <input type="checkbox"/> First aid equipment <input type="checkbox"/> Insect repellent  <hr/> <hr/>

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.



# Tetra Tech NUS, Inc.

PROJECT: \_\_\_\_\_

JOB #: \_\_\_\_\_

LOCATION: \_\_\_\_\_

DATE: \_\_\_\_\_

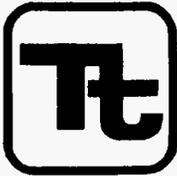
PROJECT MANAGER: \_\_\_\_\_

FOL: \_\_\_\_\_

<b>DAILY ACTIVITIES CHECKLIST</b>			
<b>Startup Checklist</b>			
<b>Activity</b>	<b>Yes</b>	<b>No</b>	<b>N/A</b>
Pertinent site activities/information entered into site logbook	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
All onsite personnel listed in logbook	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Required medical information onsite for all workers (TtNUS and Subcontractors)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Required MSDS's onsite	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Proper equipment calibrations performed (list equipment)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
1 _____	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
2 _____	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
3 _____	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
4 _____	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Calibration logs filled out	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Tailgate H&S meeting held prior to beginning field activities	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Required work permits filled out/signed	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Required utility clearances obtained	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Required PPE onsite and in use	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Information required to be posted is in place (OSHA poster, hospital route, key phone numbers, etc.)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<b>Exit Checklist</b>			
<b>Activity</b>	<b>Yes</b>	<b>No</b>	<b>N/A</b>
Logbooks completely and comprehensively filled out	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Field forms complete and accounted for/properly filed	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Samples properly packaged/shipped	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
COCs faxed to appropriate in-house personnel	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
All equipment accounted for, on charge if needed, and properly secured	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
All personnel accounted for	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Arrangements made for upcoming work (permits, clearances, equipment, etc.)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Site properly secured	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.





TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number	CT-04	Page	1 of 6
Effective Date	09/03	Revision	1
Applicability	Tetra Tech NUS, Inc.		
Prepared	Risk Assessment Department		
Approved	D. Senovich <i>ds</i>		

Subject  
SAMPLE NOMENCLATURE

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## 1.0 PURPOSE

The purpose of this document 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 data base 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 procedure shall be used consistently for all projects requiring electronic data.

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES

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

**Project Manager** - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure 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 sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

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

## 5.0 PROCEDURES

### 5.1 Introduction

The sample identification (ID) system can consist of as few as 8 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 lab 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

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Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

<b>A or N</b> <b>3- or 4-Characters</b>	<b>AAA</b> <b>2- or 3-Characters</b>	<b>A or N</b> <b>3- to 6-Characters</b>	<b>NNNN</b> <b>4-Characters</b>
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

<b>A or N</b> <b>3- or 4-Characters</b>	<b>AAA</b> <b>2- or 3-Characters</b>	<b>A or N</b> <b>3- to 6-Characters</b>	<b>NN</b> <b>2-Characters</b>	<b>-A</b>
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

<b>A or N</b> <b>3- or 4-Characters</b>	<b>AAA</b> <b>2- or 3-Characters</b>	<b>A or N</b> <b>3- to 6-Characters</b>	<b>AA</b> <b>2-Characters</b>	<b>NNN</b> <b>3-Characters</b>
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

## 5.2 Sample Identification Field Requirements

The various fields in the sample ID will 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

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 since many facilities/sites have multiple individual sites, SWMUs, operable units, 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

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 4 of 6
	Revision 1	Effective Date 09/03

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken 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 Number 1
- 125 - Solid Waste Management Unit Number 125
- 000 - Base or Facility Wide Sample (e.g., upgradient well)
- BBG - Base Background

The examples cited are only suggestions. Each Project Manager (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
- 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

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- 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.

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 designation given was 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".

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	Revision 1	Effective Date 09/03

### 5.5 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

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

<b>AA</b>	<b>NNNNNN</b>	<b>NN</b>	<b>-F</b>
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

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

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

### 5.6 Examples of Field QA/QC Sample Nomenclature

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

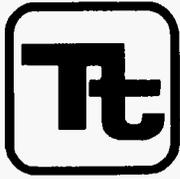
The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

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

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

### 6.0 **DEVIATIONS**

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



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number CT-05	Page 1 of 7
Effective Date 01/29/01	Revision 2
Applicability Tetra Tech NUS, Inc.	
Prepared Management Information Systems Department	
Approved D. Senovich <i>[Signature]</i>	

Subject  
DATABASE RECORDS AND QUALITY ASSURANCE

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## 1.0 PURPOSE

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

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

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

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

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

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

## 4.0 RESPONSIBILITIES

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

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

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

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

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

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

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

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

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

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

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

## 5.0 PROCEDURES

### 5.1 Introduction

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

### 5.2 File Establishment

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

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

### 5.3 Electronic Deliverables

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

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#### 5.4 Sample Tracking Forms

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

#### 5.5 Chain-of-Custody Forms

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

#### 5.6 Data Validation Letters

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

#### 5.7 Historical Data

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

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

## 6.0 RECORDS

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

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

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

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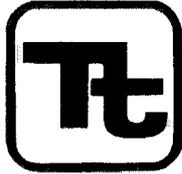
**ATTACHMENT A**



**MIS REQUEST FORM**

Tetra Tech NUS, Inc.

Project Name:	Request Date:
CTO:	Date Data Available for Production:
Project Manager:	Request in Support of:
Requestor:	Database Lead:
Program/Client:	GIS Lead:
State/EPA Region:	Statistics Lead:
	Risk Lead:
Site Name(s) (Area, OU, etc.):	
Sampling Date(s):	
Matrix: <input type="checkbox"/> GW <input type="checkbox"/> SO <input type="checkbox"/> SD <input type="checkbox"/> SW <input type="checkbox"/> Other:	
<b>Labels:</b> <input type="checkbox"/> Labels needed for an upcoming sampling event <span style="float:right">Total # of Samples</span>	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	
FOL	
<b>Data Entry:</b>	
<input type="checkbox"/> Chemical data needs to be entered from hardcopy	Estimated # of Samples
<input type="checkbox"/> Chemical data needs to be formatted electronically	
<input type="checkbox"/> Field analytical data needs to be entered from hardcopy	
<input type="checkbox"/> Geologic data needs to be entered from hardcopy	
<input checked="" type="checkbox"/> Hydrology data needs to be entered from hardcopy	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	
<b>Tables:</b>	
<input type="checkbox"/> Full Data Printout	
<input type="checkbox"/> Summary of Positive Hits	
<input type="checkbox"/> Occurance and Distribution <input type="checkbox"/> with criteria	
<input type="checkbox"/> Sampling Analytical Summary:	
<input type="checkbox"/> Other:	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	
<b>GIS:</b>	
<input type="checkbox"/> General Facility Location	
<input type="checkbox"/> Site Location	
<input type="checkbox"/> Potentiometric Contours/Groundwater Flow	
<input type="checkbox"/> Sample Location Proposed	
<input type="checkbox"/> Sample Location Existing	
<input type="checkbox"/> Tag Map Single Round	
<input type="checkbox"/> Tag Map Multiple Round	
<input type="checkbox"/> Isoconcentrations	
<input checked="" type="checkbox"/> Chart Map	
<input type="checkbox"/> 3D Visualization	
<input type="checkbox"/> EGIS CD	
<input type="checkbox"/> Other:	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	
<b>Statistics:</b> <input type="checkbox"/> Yes	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	
<b>Geostatistics:</b> <input type="checkbox"/> Yes	
Estimated Hours	Additional Instructions:
Due Date	
Complete ETS Charge No.	



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  
BOREHOLE AND SAMPLE LOGGING

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## 1.0 PURPOSE

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES

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

## 5.0 PROCEDURES

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

### 5.1 Materials Needed

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

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

### 5.2 Classification of Soils

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



FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)		FINE-GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size		COARSE-GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	
GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION
OH	Very high plasticity	OH	Highly organic clays with plasticity index greater than 16 and liquid limit greater than 50	OH	Highly organic clays with plasticity index greater than 16 and liquid limit greater than 50
CH	High plasticity	CH	Clays with plasticity index greater than 16 and liquid limit greater than 25	CH	Clays with plasticity index greater than 16 and liquid limit greater than 25
CI	Intermediate plasticity	CI	Clays with plasticity index greater than 16 and liquid limit between 25 and 50	CI	Clays with plasticity index greater than 16 and liquid limit between 25 and 50
ML	Low plasticity	ML	Clays with plasticity index less than 16 and liquid limit between 25 and 50	ML	Clays with plasticity index less than 16 and liquid limit between 25 and 50
OL	Very low plasticity	OL	Clays with plasticity index less than 16 and liquid limit less than 25	OL	Clays with plasticity index less than 16 and liquid limit less than 25

**DENSITY OF GRANULAR SOILS**

TERMINOLOGY	SYMBOL FOR PENETROMETER	BLANKET FOOT
Very Loose	1-2	
Loose	1-3	
Medium-Low	10-11	
Medium	14-17	
Very Dense	Over 20	

**CONSISTENCY OF COHESIVE SOILS**

CONSISTENCY	UNIFORMITY COEFFICIENT (U)	LIQUIDITY INDEX (LI)	FLUIDITY INDEX (FI)	FIELD IDENTIFICATION
Very Soft	Less than 1.2	Less than 1.2	Less than 1.2	Very soft to very loose
Soft	1.2 to 1.5	1.2 to 1.5	1.2 to 1.5	Soft to medium
Medium Soft	1.5 to 1.9	1.5 to 1.9	1.5 to 1.9	Medium soft to medium
Stiff	1.9 to 2.6	1.9 to 2.6	1.9 to 2.6	Stiff to medium
Very Stiff	2.6 to 4.0	2.6 to 4.0	2.6 to 4.0	Very stiff to medium
Hard	Over 4.0	Over 4.0	Over 4.0	Hard to very hard

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)		ROCK BROKENNESS	
TERMINOLOGY	SYMBOL	TERMINOLOGY	SYMBOL
Very Soft	1-2	Very soft	1-2
Soft	3-4	Soft	3-4
Medium Soft	5-7	Medium soft	5-7
Medium	8-10	Medium	8-10
Hard	11-14	Hard	11-14
Very Hard	Over 14	Very hard	Over 14

LEGEND:

- 1" - 3" Standard Sample
- 3" - 6" Standard Sample
- 6" - 12" Standard Sample
- Other Sample Specifications

LEGEND:

- 1" - 3" Standard Sample
- 3" - 6" Standard Sample
- 6" - 12" Standard Sample
- Other Sample Specifications

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### 5.2.1 USCS Classification

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

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

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inch $\Phi$ -1/2 inch $\Phi$ )" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

### 5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

### 5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

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Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

#### 5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

## FIGURE 2

## CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

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Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

#### **5.2.5 Moisture**

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

#### **5.2.6 Stratification**

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

#### **5.2.7 Texture/Fabric/Bedding**

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

#### **5.2.8 Summary of Soil Classification**

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

## FIGURE 3

## BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

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### 5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ( $\text{CaCO}_3$ ). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

#### 5.3.1 **Rock Type**

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

**FIGURE 4****GRAIN SIZE CLASSIFICATION FOR ROCKS**

<b>Particle Name</b>	<b>Grain Size Diameter</b>
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

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### 5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

### 5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

### 5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the words "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

### 5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

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The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD  
(After Deere, 1964)

$$RQD \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

### 5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

### 5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

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### 5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

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#### 5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C - Coarse	Lt - Light	Yl - Yellow
Med - Medium	BR - Broken	Or - Orange
F - Fine	BL - Blocky	SS - Sandstone
V - Very	M - Massive	Sh - Shale
Sl - Slight	Br - Brown	LS - Limestone
Occ - Occasional	Bl - Black	Fgr - Fine-grained
Tr - Trace		

#### 5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

##### 5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.
- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.
- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.
- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.
- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5  
COMPLETED BORING LOG (EXAMPLE)



BORING LOG

PROJECT NAME: NSB - SITE BORING NUMBER: SB/MW1  
 PROJECT NUMBER: 9594 DATE: 3/8/96  
 DRILLING COMPANY: SOILTEST CO. GEOLOGIST: SJ CONTI  
 DRILLING RIG: CME-55 DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			U S C S *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 e 0800	0.0 2.0	7 6 10	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME ROCK FR. - TR BRICKS (FILL)	SM	MOIST SL. ORG. ODOR FILL TO 4'±	5	0	0	0
S-2 e 0810	4.0 6.0	5 7 8	2.9/2.0	4.0	M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
S-3 e 0820	8.0 10.0	6 8 17 16	1.9/2.0	7.0 8.0	DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER: 7'±	0	0	0	0
S-4 e 0830	12.0 14.0	7 6 8	1.6/2.0	12.0	STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	5	-	-
	15.0			15.0					AUGER REF @ 15'				
	16.0			16.0	M HARD	BRN	SILTSTONE	VER	WEATHERED				
	17.0			17.0					LO *JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
	18.0			18.0					LOSING SOME				
	19.0			19.0	HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H <sub>2</sub> O @ 17'± SET TEMP 6" CAS TO 15.5				
	20.0			20.0									
	21.0			21.0					SET 2"Ø PVC SCREEN 16-25	0	0	0	0
	22.0			22.0					SAND 14-25				
	23.0			23.0					PELLETS 12-14				

\* When rock coring, enter rock brokenness.  
 \*\* Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.  
 Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ± • 1-20Z  
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z 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

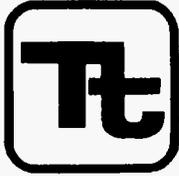
Unified Soil Classification System (USCS).

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

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT

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## 1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

## 2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

## 3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

## 4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

## 5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

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## 5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

## 5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

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previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

### **5.3 Water Level (Hydraulic Head) Measurements**

#### **5.3.1 General**

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

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Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

### 5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

### 5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.
2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):
  - Well number.
  - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
  - Time and day of the measurement.
  - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

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### 5.3.4 Water Level Measuring Devices

#### Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

#### Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "popping" or "plopping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

#### Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

#### Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

### 5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

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### 5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

### 5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

### 5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

### 6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.







TETRA TECH

# STANDARD OPERATING PROCEDURES

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Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject  
GROUNDWATER SAMPLE ACQUISITION AND  
ONSITE WATER QUALITY TESTING

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## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

## 2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

## 3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

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Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol (<sup>0</sup>/<sub>00</sub>) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

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- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

## 5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

**Knee injuries** – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

**Slips, Trips, and Falls** – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

**Cuts and Lacerations** – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.

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- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

**Vehicular and Foot Traffic Hazards** – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- **Face Traffic.** Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

## 6.0 PROCEDURES

### 6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

#### **CAUTION**

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

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Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

**CAUTION**

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.
2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.
3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

**CAUTION**

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:
  - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.
  - While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

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- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

## 6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
  - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
  - pH Paper
  - Camera and film (if appropriate)
  - Appropriate keys (for locked wells)
  - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.
- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

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### 6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons  $V = (0.163)(T)(r^2)$

where: V = Static volume of well in gallons.  
T = Linear feet of water in the well.  
r = Inside radius of well casing in inches.  
0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

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## 6.4 Evacuation of Static Water – Purging

### 6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

### 6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

#### Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

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Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

#### Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

#### Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

#### Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.

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- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

#### Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

#### Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

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To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

#### Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

#### 6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters

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used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

### 6.5.1 Measurement of pH

#### 6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

#### 6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

#### 6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)

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- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

#### 6.5.1.4 Measurement Techniques for Field Determination of pH

##### pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

##### pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

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2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

### **6.5.2 Measurement of Specific Conductance**

#### **6.5.2.1 General**

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

#### **6.5.2.2 Principles of Equipment Operation**

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

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### 6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

### 6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

## 6.5.3 Measurement of Temperature

### 6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

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### 6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

### 6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.
2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.
2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.
3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

## 6.5.4 Measurement of Dissolved Oxygen

### 6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

### 6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

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the two metals, reduction of oxygen to hydroxide ion (OH<sup>-</sup>) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

#### 6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

#### 6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

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7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.
8. Rinse the probe with deionized water.
9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

### **6.5.5 Measurement of Oxidation-Reduction Potential**

#### **6.5.5.1 General**

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

#### **6.5.5.2 Principles of Equipment Operation**

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

#### **6.5.5.3 Equipment**

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential**

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

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2. Thoroughly rinse the electrode with deionized water.
3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

### **6.5.6 Measurement of Salinity**

#### **6.5.6.1 General**

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

#### **6.5.6.2 Principles of Equipment Operation**

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

#### **6.5.6.3 Equipment**

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.6.4 Measurement Techniques for Salinity**

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the meter before going into the field.

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3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.
5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the probes with deionized water.

### **6.5.7 Measurement of Turbidity**

#### **6.5.7.1 General**

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

#### **6.5.7.2 Principles of Equipment Operation**

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

#### **6.5.7.3 Equipment**

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

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#### 6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

## 6.6 Sampling

### 6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

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Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

#### **6.6.2 Sampling Methods as Related to Low-Flow Sampling**

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:
  - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
  - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
  - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

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during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.
4. Calculate volume of well water to be removed as described in Section 6.3.
5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.
6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.
7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.
8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.
10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.
11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.
12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

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occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.
14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.
15. Process sample containers as described in SOP SA-6.1.
16. Decontaminate equipment as described in SOP SA-7.1.

## **6.7 Low-Flow Purging and Sampling**

### **6.7.1 Scope and Application**

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

### **6.7.2 Equipment**

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

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- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

### 6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).
2. Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.
3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.
4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well
5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

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6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.
  7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
  8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.
  9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.
  10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.
  11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:
    - pH  $\pm 0.2$  standard units
    - Specific conductance  $\pm 10\%$
    - Temperature  $\pm 10\%$
    - Turbidity less than 10 NTUs
    - DO  $\pm 10\%$
  12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.
- NOTE:** VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.
13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

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- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.
- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.
- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.
- Prepare samples for shipping as per SOP SA-6.1.

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**ATTACHMENT A**  
**PURGING EQUIPMENT SELECTION**

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

**ATTACHMENT A**  
**PURGING EQUIPMENT SELECTION**  
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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/L length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	\$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	\$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefze®	0-100	0-500 mL/min depending on lift	\$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	\$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	\$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	\$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	\$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	\$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	\$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	\$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	\$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	\$300-400	Requires compressed gas; piezometric level indicator; other materials available.

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**PURGING EQUIPMENT SELECTION**  
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Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	\$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	\$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	\$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	\$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	\$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	\$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	\$800-1,000	Compressed gas required; DC control module; custom built.

## Construction Material Abbreviations:

PE Polyethylene  
 PP Polypropylene  
 PVC Polyvinyl chloride  
 SS Stainless steel  
 PC Polycarbonate  
 EPDM Ethylene-propylene diene (synthetic rubber)

## Other Abbreviations:

NA Not applicable  
 AC Alternating current  
 DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

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# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T. E. Johnston</i>		

Subject  
SURFACE WATER AND SEDIMENT SAMPLING

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## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

## 2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

## 3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

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custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

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

## 5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

**Bridge/Boat Sampling** – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

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Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United States Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.
- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

**Entering Water to Collect Samples** – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.
- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.
- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.
- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.
- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.
- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.
- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

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Working in these areas, also recognize the following hazards and means of protection against them:

**Insects** are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellent. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

**Safety Reminder**

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

**Poisonous Plants** – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

**Temperature-Related Stress** – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

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- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.
- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder*).

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

## 6.0 PROCEDURES

### 6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

#### 6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.

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4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

**Safety Reminder**

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
- Required decontamination equipment.
- Required sample containers.
- Sealable polyethylene bags (e.g., Ziploc<sup>®</sup> baggies).
- Heavy-duty cooler.
- Ice.

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- Paper towels and garbage bags.
- Chain-of-custody records and custody seals.

#### Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.
2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.
3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.
4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.
5. Use a clean paper towel to clean and dry the outside of the container.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

#### **CAUTION**

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

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### Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.
3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
4. Raise the sampler and cap the bottle.
5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

### Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.
2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.
3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).
4. Use a paper towel to clean and dry the outside of the container.
5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

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### 6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.
- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.
- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.
- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.
- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

### 6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

### 6.3 Sediment Sampling

#### 6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

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respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

### 6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

#### **SAFETY REMINDER**

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices
- A minimum of three people are required if any of the following conditions are anticipated or observed:
  - Work in a waterway that is turbulent or swift that could sweep a sampler down stream should he or she fall in accidentally.
  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.
  - Waterway is tidal, and conditions such as those listed above could rapidly change.

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The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

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### Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.
2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

### Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.
2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.
3. Open the sampler to retrieve the sediment.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.

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6. Use a paper towel to clean and dry the outside of each container.
7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

***SAFETY REMINDER***

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.
2. Cap the top of the tube to provide suction and reduce the chance of losing the sample.
3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.
6. Use a paper towel to clean and dry the outside of each container.
7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

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All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

## 7.0 REFERENCES

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U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

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**APPENDIX C  
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION**

**C.1 Defining the Sampling Program**

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

**C.1.1 Sampling Program Objectives**

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

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### **C.1.2 Location of Sampling Stations**

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

### **C.1.3 Frequency of Sampling**

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

## **C.2 Surface Water Sample Collection**

### **C.2.1 Streams, Rivers, Outfalls and Drainage Features**

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

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Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

### **C.2.2 Lakes, Ponds and Reservoirs**

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

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### C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.
- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.
- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Effective Date	09/03	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>DS</i>		

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)

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## 1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

## 2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

## 3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

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Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

## **5.0 SOIL SAMPLING PROCEDURES**

### **5.1 General**

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

### **5.2 Sampling Equipment**

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

### **5.3 DPT Sampling Methodology**

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.
- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.
- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.
- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.

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- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

## 6.0 GROUNDWATER SAMPLING PROCEDURES

### 6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

### 6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

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### 6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.
- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.
- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ( $\pm 10$  percent), the well may be sampled.
- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.
- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).
- Decontaminate the equipment before moving to the next location.

### 7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

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**ATTACHMENT 1  
SAFE WORK PERMIT FOR DPT OPERATIONS**

Permit No. \_\_\_\_\_ Date: \_\_\_\_\_ Time: From \_\_\_\_\_ to \_\_\_\_\_

**SECTION I: General Job Scope**

- I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**
- II. Required Monitoring Instruments: \_\_\_\_\_
- III. Field Crew: \_\_\_\_\_
- IV. On-site Inspection conducted  Yes  No Initials of Inspector \_\_\_\_\_

TtNUS

**SECTION II: General Safety Requirements (To be filled in by permit issuer)**

- |  |  |  |
|--|--|--|
| V. Protective equipment required   | 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"/>
Emergency alarms.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Evacuation routes.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Assembly points.....	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

IX. Site Preparation

- Utility Clearances obtained for areas of subsurface investigation  Yes  No
- Physical hazards removed or blockaded  Yes  No
- Site control boundaries demarcated/signage  Yes  No

X. Equipment Preparation

- |  |                              |  |
|--|------------------------------|--|
| Equipment drained/depressurized.....                       | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Equipment purged/cleaned.....                              | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Isolation checklist completed.....                         | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Electrical lockout required/field switch tested.....       | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Blinds/misalignments/blocks & bleeds in place.....         | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Hazardous materials on walls/behind liners considered..... | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |

- XI. Additional Permits required (Hot work, confined space entry).....  Yes  No  
*If yes, complete permit required or contact Health Sciences, Pittsburgh Office*

XII. Special instructions, precautions:

\_\_\_\_\_

\_\_\_\_\_

Permit Issued by: \_\_\_\_\_ Permit Accepted by: \_\_\_\_\_



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Effective Date 04/072008	Revision 9
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/O<sub>2</sub> meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

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- Soil type classification

## 7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

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

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



Tetra Tech NUS, Inc.

**SOIL & SEDIMENT SAMPLE LOG SHEET**

Page \_\_\_ of \_\_\_

Project Site Name: _____	Sample ID No.: _____
Project No.: _____	Sample Location: _____
<input type="checkbox"/> Surface Soil	Sampled By: _____
<input type="checkbox"/> Subsurface Soil	C.O.C. No.: _____
<input type="checkbox"/> Sediment	Type of Sample:
<input type="checkbox"/> Other: _____	<input type="checkbox"/> Low Concentration
<input type="checkbox"/> QA Sample Type: _____	<input type="checkbox"/> High Concentration

GRAB SAMPLE DATA:			
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

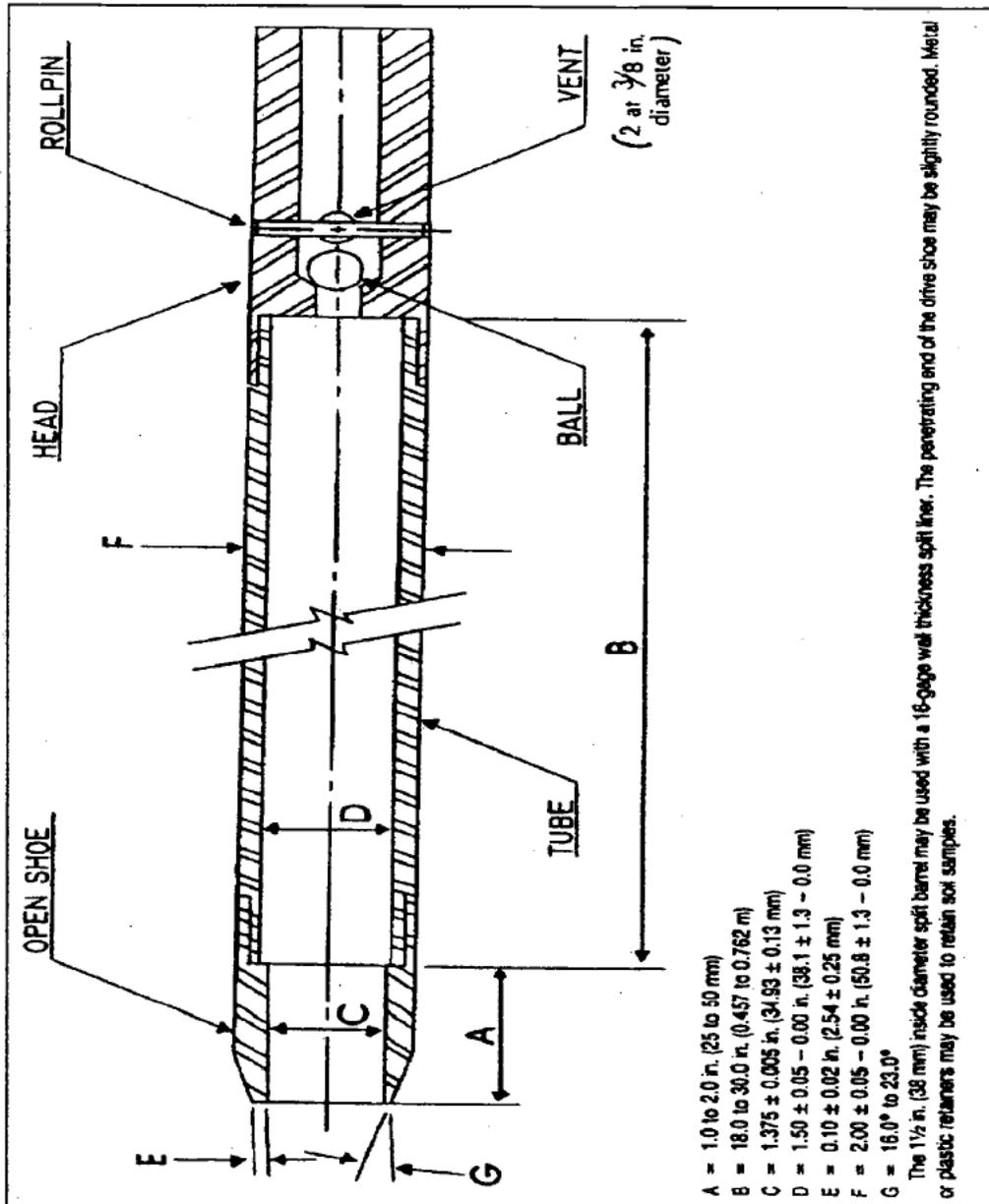
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

<b>OBSERVATIONS / NOTES:</b>	<b>MAP:</b>

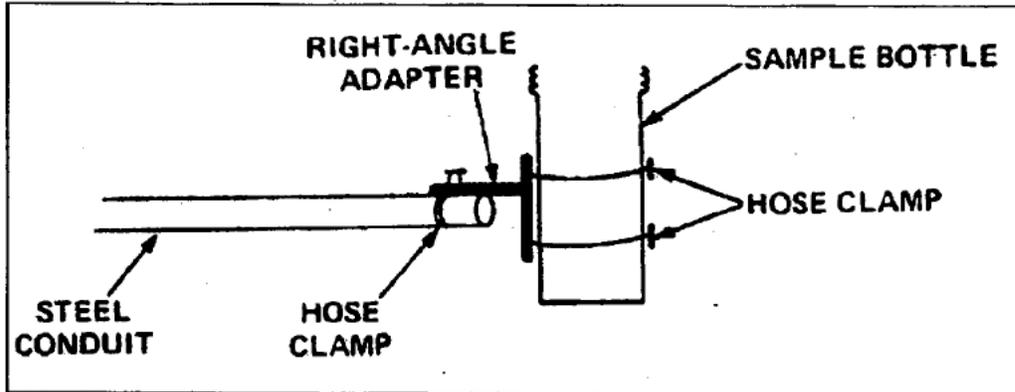
<b>Circle if Applicable:</b>	<b>Signature(s):</b>
MS/MSD      Duplicate ID No.:	

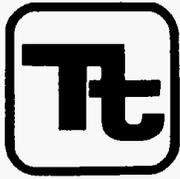
### ATTACHMENT B SPLIT-SPOON SAMPLER





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





TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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 - H<sub>2</sub>SO<sub>4</sub>
- Nitric Acid - HNO<sub>3</sub>
- Sodium Hydroxide - NaOH

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#### Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>

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 HNO<sub>3</sub>, 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 <sub>2</sub> SO <sub>4</sub> )	1 part concentrated H <sub>2</sub> SO <sub>4</sub> : 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO <sub>3</sub> )	Undiluted concentrated HNO <sub>3</sub>	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 <sup>(1)</sup>	Sample Size	Preservation <sup>(2)</sup>	Holding Time <sup>(2)</sup>
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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 <sup>(9)</sup>
	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 <sub>3</sub> 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 <sup>(1)</sup>	Preservation <sup>(2)(3)</sup>	Maximum Holding Time <sup>(4)</sup>
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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 <sub>2</sub> SO <sub>4</sub> 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 <sub>2</sub> SO <sub>4</sub> 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 <sup>(5)</sup>	14 days <sup>(6)</sup>
Fluoride	P	None required	28 days
Hardness	P, G	HNO <sub>3</sub> to pH 2; H <sub>2</sub> SO <sub>4</sub> to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H <sub>2</sub> SO <sub>4</sub> 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 <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> 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 <sup>(1)</sup>	Preservation <sup>(2)(3)</sup>	Maximum Holding Time <sup>(4)</sup>
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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:<sup>(7)</sup>**

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

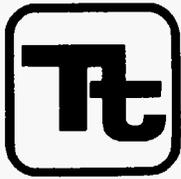
**ORGANIC TESTS:<sup>(8)</sup>**

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



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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

Subject  
FIELD DOCUMENTATION

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

## 2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

## 3.0 GLOSSARY

None

## 4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

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

## 5.0 PROCEDURES

### 5.1 Site Logbook

#### 5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

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

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

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

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

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

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

### **5.1.2 Photographs**

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

### **5.2 Field Notebooks**

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

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### 5.3 **Field Forms**

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (<http://intranet.ttnus.com>) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

#### 5.3.1 **Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results**

##### 5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

##### 5.3.1.2 Sample Label

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

##### 5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

##### 5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

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#### 5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

### 5.3.2 **Hydrogeological and Geotechnical Forms**

#### 5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

#### 5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

#### 5.3.2.3 Packer Test Report Form

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

#### 5.3.2.4 Boring Log

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

#### 5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

#### 5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

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#### 5.3.2.7 Miscellaneous Monitoring Well Forms

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

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

#### 5.3.2.8 Miscellaneous Field Forms - QA and Checklists

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

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

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

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

### 5.3.3 **Equipment Calibration and Maintenance Form**

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

### 5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

#### 5.4.1 **Daily Activities Report**

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

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#### 5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

#### 5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

#### 5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

### 5.4.2 **Weekly Status Reports**

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

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

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

### 6.0 **LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS**

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

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

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**ATTACHMENT A  
TYPICAL SITE LOGBOOK ENTRY**

START TIME: \_\_\_\_\_ DATE: \_\_\_\_\_

SITE LEADER: \_\_\_\_\_

PERSONNEL: \_\_\_\_\_

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well \_\_\_\_ resumes. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well \_\_\_\_\_.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well \_\_\_\_\_.
4. Well \_\_\_\_\_ drilled. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 2, page \_\_\_\_ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well \_\_\_\_\_ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit \_\_\_\_\_.
8. Test pit \_\_\_\_\_ dug with cuttings placed in dump truck. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit \_\_\_\_ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

\_\_\_\_\_  
Field Operations Leader

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**ATTACHMENT B**

	Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
			Site:
		Location:	
Sample No:		Matrix:	
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	



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ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

<u>Signature</u> <hr/> <u>Date</u> <hr/> <b>CUSTODY SEAL</b>		<b>CUSTODY SEAL</b> <hr/> <u>Date</u> <hr/> <u>Signature</u>
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# STANDARD OPERATING PROCEDURES

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Effective Date 01/28/2009	Revision 6
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

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## 1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment, chemical sampling equipment and field operation and analytical equipment.

## 2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

## 3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent - A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

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#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

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

#### 5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

#### 6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

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- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

## 7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

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- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

## 7.1 Decontamination Pad Design/Construction Considerations

### 7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:
  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

The selected decontamination site should include the following, where possible:

- Areas where potable water and electricity are provided.

#### **Safety Reminder**

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).
- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

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- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.
- Areas that offer sufficient size to carry out the specific decontamination sequence.
- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:
  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.
  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.
  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.
  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.
  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:
  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

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- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.
- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

### **7.1.2 Decontamination Activities at Drill Rigs/DPT Units**

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

### **7.1.3 Decontamination Activities at Remote Sample Locations**

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

## **7.2 Equipment Decontamination Procedures**

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

### **7.2.1 Monitoring Well Sampling Equipment**

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.
2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.
3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.
4. Remove the pump and tubing/bailer from the container
5. Rinse external pump components using tap water.

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6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

**CAUTION**

Do not rinse PE, PVC, and associated tubing with solvents –  
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.
8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.
9. Drain residual deionized water to the extent possible.
10. Allow components of the equipment to air dry.
11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.
12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

**SAFETY REMINDER**

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

**NOTE**

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

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### 7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.
- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:
  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

### 7.2.2 **Downhole Drilling Equipment**

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

**CAUTION**  
 Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

**CAUTION**  
 In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

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4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol
5. To the extent possible, allow components to air dry.
6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.
7. Wrap or cover equipment in clear plastic until it is time to be used.

**SAFETY REMINDER**

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.
2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.
3. Always wear PPE as specified in the HASP such as:
  - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.
4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.
5. Do not modify equipment unless the manufacturer has approved the modifications.

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### 7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

**CAUTION**

Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

**CAUTION**

When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

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### 7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

#### 7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

**NOTE**

Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

**NOTE**

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).
4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:
  - Enclose areas accessible by the general public using construction fencing and signs.
  - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
  - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
  - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
  - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
  - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

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- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

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**CAUTION**

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

**7.4 Decontamination Evaluation**

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.
- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

**NOTE**

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:
  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.
  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

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**NOTE**

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.



# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject DECONTAMINATION OF FIELD EQUIPMENT

## Attachment A iDW Label

**INVESTIGATION DERIVED WASTE**

GENERATOR INFORMATION:

SITE \_\_\_\_\_ JOB NO. \_\_\_\_\_

LOCATION \_\_\_\_\_

DATE \_\_\_\_\_

DRUM# \_\_\_\_\_

CONTENTS \_\_\_\_\_

VOLUME \_\_\_\_\_

CONTACT \_\_\_\_\_

EMERGENCY PHONE NUMBER \_\_\_\_\_

## **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.

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- 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**

<b>Construction Material</b>	<b>Analyte Group Sampled</b>	<b>SOP Reference</b>	<b>Base of Operations</b>	<b>On-Site</b>
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 <sup>1</sup> 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

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<sup>i</sup> 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 <b>was not</b> 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><b>DECONTAMINATION SUPPLIES</b></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><b>REFERENCE MATERIALS</b></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><b>HEALTH &amp; SAFETY SUPPLIES</b></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><b>SAMPLE TRANSPORTATION SUPPLIES</b></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><b>FIELD MEASUREMENT EQUIPMENT</b></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><b>PRESERVATION SUPPLIES</b></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><b>DOCUMENTATION SUPPLIES</b></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: \_\_\_\_\_

**WASTE**

- Stainless steel scoop
- Stainless steel spoons or spatulas
- Stainless steel push tubes
- Stainless steel auger
- Stainless steel Ponar dredge
- Glass coliwasa
- Drum thief
- Mucksucker
- Dipstick
- Stainless steel bacon bomb
- Stainless steel bailer
- Teflon bailer
- Peristaltic pump
- Stainless steel split spoon
- Roto-hammer
- Glass tubing

**SHELLFISH**

- Seine
- Trawl
- Bucket type/double pole
- Tong/Double handed grab
- Line or cable operated grab bucket
- Petersen
- Ponar
- Ekman
- Orange-peel grab
- Biological or hydraulic dredge
- Scoops/shovels
- Scrapers
- Rakes
- D-traps
- Processing Equipment
  - Holding trays
  - Stainless steel shucking knife
  - Calipers or ruler
  - Aluminum foil
  - Plastic bags

**FINFISH**

- Electrofishing devices
- Seines

- Trawls
- Angling
- Gill net
- Trammel net
- Hoop, fyke & pound nets
- D-traps
- Processing Equipment
  - Holding trays
  - Measuring board or ruler
  - Stainless steel descaler
  - Stainless steel scalpel
  - Balance
  - Aluminum foil
  - Plastic bags

**BIOLOGICAL COMMUNITY**

**SAMPLING**

- Phytoplankton
  - Van Dorn
  - Alpha bottle
  - Logol's solution
- Periphyton
  - Periphytometer
  - Microscope slides
  - 100% buffered formalin
  - Nylon twine
- Qualitative Periphyton Sampling
  - Stainless steel spatula/spool
  - Stainless steel forceps
  - Suction bulb
  - Preservative
    - Buffered formalin
    - Lugol's solution
  - M3
  - Resealable plastic bags
  - White picking pan
- Benthic Macroinvertebrates
  - Forceps
  - Transfer pipettes
  - White picking pans
  - 10X hand lens
  - Alcohol-filled jars
  - Dip net (30 mesh)
  - Hester-Dendy
  - Coring device

- Dredge
  - Ekman
  - Petite ponar
  - 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., <math><6^{\circ}\text{C}</math>), 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 <sup>1</sup>	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. <sup>2</sup>
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. <sup>2</sup> 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 <b>if</b> appropriate liners are used. Use Teflon liners for organics. Use plastic or Teflon liners for metals. Do not use if weathered, corroded or pitted. <sup>2</sup> 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. <sup>2</sup>
Plastics <sup>3</sup>		
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.

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

<b>Construction Material<sup>1</sup></b>	<b>Acceptable Analyte Groups</b>	<b>Precautions</b>
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. <sup>4</sup>	Minimize contact with sample. Use only if no alternative material exists.
<b>Glass</b>		
Glass, borosilicate	All analyte groups except silica and boron.	

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

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<sup>1</sup> 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.)

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

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

<sup>4</sup> 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**

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

**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> <sup>1</sup>	<u>TUBING</u>			
<b>WATER SAMPLING</b>					
<b>GROUNDWATER</b>					
1 Positive displacement pumps <sup>2</sup>					
a. Submersible (turbine, helical rotor, gear driven)	SS, Teflon	SS, Teflon, PE, PP	Purging	All analyte groups	<sup>3,4,5</sup> ; must be variable speed
			Sampling	All analyte groups	<sup>3,4,5</sup> must be variable speed
	SS, Teflon	Non-inert <sup>6</sup>	Purging	All analyte groups	<sup>3,4,5</sup> must be variable speed; polishing required <sup>7</sup>
			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 <sup>6</sup>	Non-inert <sup>6</sup>	Purging	All analyte groups	<sup>3,4,5</sup> must be variable speed; polishing required <sup>7</sup>
			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	<sup>3,4,5</sup> must be variable speed
			Sampling	All analyte groups	<sup>3,4</sup> 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 <sup>6</sup>	Purging	All analyte groups	<sup>3,4</sup> must be variable speed; polishing required <sup>7</sup>
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	<b>This configuration is not recommended</b> <sup>3,4</sup> must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert <sup>6</sup>	Non-inert <sup>6</sup>	Purging	All analyte groups	<sup>3,4</sup> must be variable speed; polishing required <sup>7</sup>
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	<sup>3,4</sup> must be variable speed; polishing required <sup>7</sup> 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> <sup>1</sup>	<u>TUBING</u>			
<b>2. Suction lift pumps</b>					
a. Centrifugal	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	<sup>4</sup> foot-valve required Must be variable speed
	N/A	Non-inert <sup>6</sup>	Purging	All analyte groups	<sup>4</sup> foot-valve required; polishing required Must be variable speed
b. Peristaltic	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	<sup>4</sup> foot-valve required; polishing required or continuous pumping required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile organics	<sup>4</sup> Silicone tubing in pump head Must be variable speed
	N/A	Non-inert <sup>6</sup>	Purging	All analyte groups	<sup>4</sup> foot-valve required Must be variable speed
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	<sup>4</sup> Silicone tubing in pump head Must be variable speed
<b>3. Bailers</b>					
	SS, Teflon, PE, PP	N/A	Purging	All analyte groups	None; <b>not recommended</b>
		N/A	Sampling	All analyte groups	None; <b>not recommended</b>
	Non-inert <sup>6</sup>	N/A	Purging	All analyte groups <u>except</u> volatile and extractable organics	None; <b>not recommended</b> 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; <b>not recommended</b> If sampling for metals, the tubing must be non-metallic if not SS
<b><u>SURFACE WATER</u></b>					
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 <sup>6</sup>	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> <sup>1</sup>	<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 <sup>o</sup>	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 <sup>o</sup>	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 <sup>o</sup>		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> <sup>1</sup>	<u>TUBING</u>			
<b>SOLID SAMPLING</b>					
<b>SOILS</b>					
1. Core barrel (or liner)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups <sup>8</sup>	9, 10, 11
	Non-inert <sup>6</sup> nonmetallics	N/A	Sampling	All analyte groups	12
	Non-inert <sup>6</sup> 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 <sup>8</sup>	
			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 <sup>8</sup>	11
			Compositing or homogenizing	All analyte groups except volatile organics	11
	Non-inert <sup>6</sup>	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 <sup>8</sup>	None
	Non-SS	N/A	Sampling	All analyte groups <sup>8</sup>	10,11,12
5. Split spoon	SS or carbon steel w/ Teflon insert	N/A	Sampling	All analyte groups <sup>8</sup>	10,11,12
6. Shelby tube	SS	N/A	Sampling	All analyte groups <sup>8</sup>	9
	Carbon steel	N/A	Sampling	All analyte groups	9,10,12
<b>SEDIMENT</b>					
1. Coring devices	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups <sup>8</sup>	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> <sup>1</sup>	<u>TUBING</u>			
	Non-inert <sup>6</sup> nonmetallics	N/A	Sampling	All analyte groups	<sup>12</sup>
	Non-inert <sup>6</sup> metals	N/A	Sampling	All analyte groups	<sup>9,10,11</sup>
2. Grab – Young, Petersen, Shipek	Teflon, Teflon-lined, SS	N/A	Sampling	All analyte groups <sup>8</sup>	None
	Carbon steel	N/A	Sampling	All analyte groups	<sup>10,11</sup>
3. Dredges – Eckman, Ponar, Petit Ponar Van Veen	SS	N/A	Sampling	All analyte groups <sup>8</sup>	None
	Carbon steel, brass	N/A	Sampling	All analyte groups	<sup>10,11</sup>
4. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling Compositing	All analyte groups <sup>8</sup> 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 Compositing or homogenizing	All analyte groups <sup>8</sup> All analyte groups except volatile organics	<sup>11</sup>
	Non-inert <sup>6</sup>	N/A	Compositing or homogenizing	All analyte groups <u>except</u> volatile and extractable organics	none <sup>11</sup> must be nonmetallic if not SS
<b>WASTE</b> <sup>13</sup>					
Scoop	SS	N/A	Liquids, solids & sludges	All analyte groups <sup>8</sup>	Cannot collect deeper phases
Spoon	SS	N/A	Solids, sludges	All analyte groups <sup>8</sup>	Cannot collect deeper phases
Push tube	SS	N/A	Solids, sludges	All analyte groups <sup>8</sup>	Cannot collect deeper phases
Auger	SS	N/A	Solids	All analyte groups <sup>8</sup>	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> <sup>1</sup>	<u>TUBING</u>			
Sediment sampler	SS	N/A	Impoundments, piles	All analyte groups <sup>8</sup>	None
Ponar dredge	SS	N/A	Solids, sludges & sediments	All analyte groups <sup>8</sup>	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 <sup>8</sup>	Not recommended for viscous wastes
Bailer	SS, Teflon	N/A	Liquids	All analyte groups <sup>8</sup>	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 <sup>8</sup>	
Roto-Hammer	Steel	N/A	Solids	All analyte groups <sup>8</sup>	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**

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- <sup>1</sup> 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.).
- <sup>2</sup> If used to collect volatile or extractable organics, all power cords and other tubing must be encased in Teflon, PE or PP.
- <sup>3</sup> If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.
- <sup>4</sup> 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.
- <sup>5</sup> In-line check valve required.
- <sup>6</sup> "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.
- <sup>7</sup> "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.
- <sup>8</sup> Do not use if collecting for hexavalent chromium (Chromium<sup>+6</sup>)
- <sup>9</sup> 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.
- <sup>10</sup> 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.
- <sup>11</sup> Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest.
- <sup>12</sup> If non-inert-liner, core barrel or implement is used, take samples from the interior part of the collected sample.
- <sup>13</sup> 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 <sup>1</sup>	Preservation <sup>2, 3</sup>	Maximum holding time <sup>4</sup>
<b>Table IA—Bacterial Tests:</b>			
1–5. Coliform, total, fecal, and <i>E. coli</i>	PA, G	Cool, <10 °C, 0.0008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours <sup>6, 7</sup>
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours <sup>6</sup>
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours <sup>6</sup>
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours <sup>6</sup>
<b>Table IA—Aquatic Toxicity Tests:</b>			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C <sup>8</sup>	36 hours
<b>Table IB—Inorganic Tests:</b>			
1. Acidity	P, FP, G	Cool, ≤6 °C <sup>9</sup>	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C <sup>9</sup>	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours
10. Boron	P, FP, or Quartz	HNO <sub>3</sub> 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 <sup>9</sup>	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> 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 <sup>9</sup>	48 hours
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C <sup>9</sup> , NaOH to pH>12 <sup>10</sup> , reducing agent <sup>5</sup>	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> 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 <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
<b>Table IB—Metals:</b>			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C <sup>9</sup> , pH = 9.3–9.7 <sup>12</sup>	28 days
35. Mercury (CVAA)	P, FP, G	HNO <sub>3</sub> 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 <sup>1</sup>	Preservation <sup>2, 3</sup>	Maximum holding time <sup>4</sup>
35. Mercury (CVAFS)	FP, G; and FP-lined cap <sup>13</sup>	5 mL/L 12N HCl or 5 mL/L BrCl <sup>13</sup>	90 days <sup>13</sup>
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 <sub>3</sub> to pH<2, or at least 24 hours prior to analysis <sup>14</sup>	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours
41. Oil and grease	G	Cool, ≤6 °C <sup>9</sup> , HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C <sup>9</sup> , HCl, H <sub>2</sub> SO <sub>4</sub> , or H <sub>3</sub> PO <sub>4</sub> to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤6 °C <sup>9</sup>	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 <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C <sup>9</sup>	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C <sup>9</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C <sup>9</sup>	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C <sup>9</sup>	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C <sup>9</sup>	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C <sup>9</sup>	7 days
61. Silica	P or Quartz	Cool, ≤6 °C <sup>9</sup>	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C <sup>9</sup>	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C <sup>9</sup>	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C <sup>9</sup> , 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 <sup>9</sup>	48 hours

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**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 <sup>1</sup>	Preservation <sup>2, 3</sup>	Maximum holding time <sup>4</sup>
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours

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

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**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 <sup>1</sup>	Preservation <sup>2, 3</sup>	Maximum holding time <sup>4</sup>
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C <sup>9</sup>	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C <sup>9</sup>	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year
<b>Table ID—Pesticides</b>			
Tests: 1–70. Pesticides <sup>18</sup>	G, FP-lined cap	Cool, ≤6 °C <sup>9</sup> , pH 5–9 <sup>22</sup>	7 days until extraction, 40 days after extraction
<b>Table IE—Radiological Tests:</b>			
1–5. Alpha, beta, and radium	P, FP, G	HNO <sub>3</sub> to pH<2	6 months
<b>Table IH—Bacterial Tests:</b>			
1. <i>E. coli</i>			
2. Enterococci	PA, G	Cool, <10 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	6 hours <sup>6</sup>
<b>Table IH—Protozoan Tests:</b>			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours. <sup>23</sup>
9. Giardia	LDPE; field filtration	0–8 °C	96 hours <sup>23</sup>

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

<sup>1</sup> “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.

<sup>2</sup> 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,

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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).

<sup>3</sup> 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 (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 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).

<sup>4</sup> 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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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.

<sup>5</sup> 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 ( $\text{NaAsO}_2$ ), or sodium borohydride ( $\text{NaBH}_4$ ). 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  $\text{NaBH}_4$  or  $\text{NaAsO}_2$  is used, 25 mg/L  $\text{NaBH}_4$  or 100 mg/L  $\text{NaAsO}_2$  will reduce more than 50 mg/L of chlorine (see method “Kelada-01” and/or Standard Method

4500–CN<sup>-</sup> 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.

<sup>6</sup> 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.

<sup>7</sup> 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.

<sup>8</sup> 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.

<sup>9</sup> Aqueous samples must be preserved at  $\leq 6$  °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 “ $\leq 6$  °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<sup>th</sup> of 1 degree); rather, three

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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).

<sup>10</sup> 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 (S<sub>8</sub>), 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)

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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  $\mu\text{g}$  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  $\mu\text{g}$  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  $\geq 12$  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).

**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)

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

<sup>11</sup> 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.

<sup>12</sup> 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.

<sup>13</sup> 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.

<sup>14</sup> 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.

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

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

<sup>17</sup> 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.

**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)

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<sup>18</sup> 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  $\leq 6$  °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).

<sup>19</sup> If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to  $4.0 \pm 0.2$  to prevent rearrangement to benzidine.

<sup>20</sup> Extracts may be stored up to 30 days at  $< 0$  °C.

<sup>21</sup> 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

<sup>22</sup> 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%  $\text{Na}_2\text{S}_2\text{O}_3$ .

<sup>23</sup> 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 <sup>1</sup>	Container <sup>2</sup>	Preservation <sup>3</sup>	Maximum Holding Time <sup>4</sup>
Bromine	DPD Colorimetric <sup>5</sup>	SM 4500-CI-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 <sup>6</sup>	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G <sup>7</sup>	Dark 4°C Filtered, dark, 20°C	48 hours chilled until filtration <sup>8</sup> , and analyze immediately or 48 hours chilled until filtration <sup>8</sup> , and 28 days (frozen) after filtration
Corrosivity	Calculated (CaCO <sub>3</sub> Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C <sup>9</sup>	7 days <sup>9</sup>
FL-PRO	Gas Chromatography	DEP (11/1/95)	G only	Cool 4°C, H <sub>2</sub> SO <sub>4</sub> 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 <sup>10</sup> Hydrometric <sup>10</sup>	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days <sup>10</sup>
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 <sub>2</sub> SO <sub>4</sub> or HCl to pH<2	28 days
Transparency	Irradiometric <sup>11</sup>	62-302.200(6), FAC	_____	_____	Analyze in-situ
Un-ionized Ammonia	Calculated <sup>12</sup>	DEP-SOP <sup>13</sup>	P, G	Cool 4°C Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>12</sup>	8 hours unpreserved 28 days preserved <sup>12</sup>
Organic Pesticides <sup>14</sup>	GC and HPLC	EPA (600-series) <sup>14</sup>	<sup>15</sup>	<sup>15</sup>	<sup>15</sup>

<sup>1</sup> SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 20<sup>th</sup> 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.

<sup>2</sup> P = plastic, G = glass.

<sup>3</sup> When specified, sample preservation should be performed immediately upon sample collection.

<sup>4</sup> 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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- <sup>5</sup> 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).
- <sup>6</sup> 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.
- <sup>7</sup> Collect samples in opaque bottles and process under reduced light.
- <sup>8</sup> 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..
- <sup>9</sup> 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.
- <sup>10</sup> 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.
- <sup>11</sup> 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.
- <sup>12</sup> 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 H<sub>2</sub>SO<sub>4</sub> 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.
- <sup>13</sup> DEP Central Analytical Laboratory, Tallahassee, FL, Revision No. 2, 2-12-2001. The document is available from the DEP Standards & Assessment Section..
- <sup>14</sup> 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).
- <sup>15</sup> 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 <sup>1</sup>	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C <sup>1</sup> 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 <sup>1</sup>	1 month until extraction, 4 days after extraction <sup>2</sup>
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4°± 2°C <sup>1</sup>	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool 4°C <sup>1</sup>	24 hours
Aggregate Properties			Glass or plastic	Cool 4°C <sup>1</sup>	14 days
Inorganic nonmetallics all except:			Glass or plastic	Cool 4°C <sup>1</sup>	28 days
----- Sulfite, Nitrate, Nitrite & o-phosphate			Glass or plastic		48 hours
----- 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.

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

<sup>2</sup> 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 <sup>①</sup>	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 <sub>4</sub> / 4°C	5035 - Section 7.2	14 D	Any recognized VOC Method
				5035 - 6.1.1 <sup>②</sup>	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
				5035 - 6.1.1 <sup>②</sup>	4°C / -10°C <sup>③,④</sup>	5035 - Section 7.2	48 H / 14 D <sup>⑤</sup>	Any recognized VOC Method
	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 <sup>②,⑥,⑦</sup>	4°C	5035 - Section 7.2	48 H	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1 <sup>⑥,⑦</sup>	NaHSO <sub>4</sub> / 4°C	5035 - Section 7.2 <sup>⑥</sup>	48 H / 14 D <sup>⑤</sup>	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1 <sup>②⑥⑦</sup>	4°C / -10°C <sup>③,④</sup>	5035 - Section 7.2 <sup>⑥</sup>	48 H / 14 D <sup>⑤</sup>	Any recognized VOC Method
□200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3 <sup>⑥</sup>	EnCore or equivalent	5035 - 6.1.3 <sup>⑥,⑦</sup>	4°C	5035 - Sections 7.3.2 & 7.3.3 <sup>⑥</sup>	48 H / 14 D <sup>⑤</sup>	Any recognized VOC Method
□200 ug/kg <sup>®</sup>	Coring Device	5035 - Section 6.2.2.3 <sup>⑥</sup>	Glass Vial w/ PTFE-silicone Septum	6.1.3 <sup>⑥</sup>	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**

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- 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 <sup>1</sup>	Holding Time <sup>2</sup>	Holding Time for Extract <sup>3</sup>	Container <sup>4</sup>
MICROBIOLOGICAL-BACTERIA	Cool < 10°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>			P or G
Total Coliforms, fecal coliforms & <i>E. coli</i> in drinking water	Cool < 10°C <sup>6</sup> , Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	30 Hours <sup>7</sup>		P or G
Total coliforms and fecal coliforms in source water Heterotrophic bacteria in drinking water	Cool < 10°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>5</sup>	8 hours		P or G
Gross Alpha	Conc. HCl or HNO <sub>3</sub> to pH <2 <sup>8,9</sup>	6 mo		P or G
Gross beta	Conc. HCl or HNO to pH <2 <sup>8,9</sup>	6 mo		P or G
Strontium-89	Conc. HCl or HNO to pH <2 <sup>8,9</sup>	6 mo		P or G
Strontium-90	Conc. HCl or HNO to pH <2 <sup>8,9</sup>	6 mo		P or G
Radium-226	Conc. HCl or HNO to pH <2 <sup>8,9</sup>	6 mo		P or G
Radium-228	Conc. HCl or HNO to pH <2 <sup>8,9</sup>	6 mo		P or G
Cesium-134	Concentrated HCl to pH <<2 <sup>8,9</sup>	6 mo		P or G
Iodine-131	None	8 da		P or G
Tritium	None	6 months		G
Uranium	Conc. HCl or HNO <sub>3</sub> to pH <2 <sup>8,9</sup>	6 mo		P or G
Photon emitters	Conc. HCl or HNO <sub>3</sub> to pH <2 <sup>8,9</sup>	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 <sup>1</sup>	Holding Time <sup>2</sup>	Holding Time for Extract <sup>3</sup>	Container <sup>4</sup>
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 <sup>1</sup>	Holding Time <sup>2</sup>	Holding Time for Extract <sup>3</sup>	Container <sup>4</sup>
	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 <sub>2</sub> SO <sub>4</sub> 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

<sup>1</sup> Preservation, when required, must be done immediately upon sample collection.

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

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

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

<sup>5</sup> 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**

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- <sup>6</sup> 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.
- <sup>7</sup> 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.
- <sup>8</sup> 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.
- <sup>9</sup> 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><b>ANALYTE NAME</b></i>	<i><b>CONTAINER</b></i>	<i><b>PRESERVATION</b></i>	<i><b>MAX HOLDING TIME</b></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**

<b>Holding Time (Days)</b>				
	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

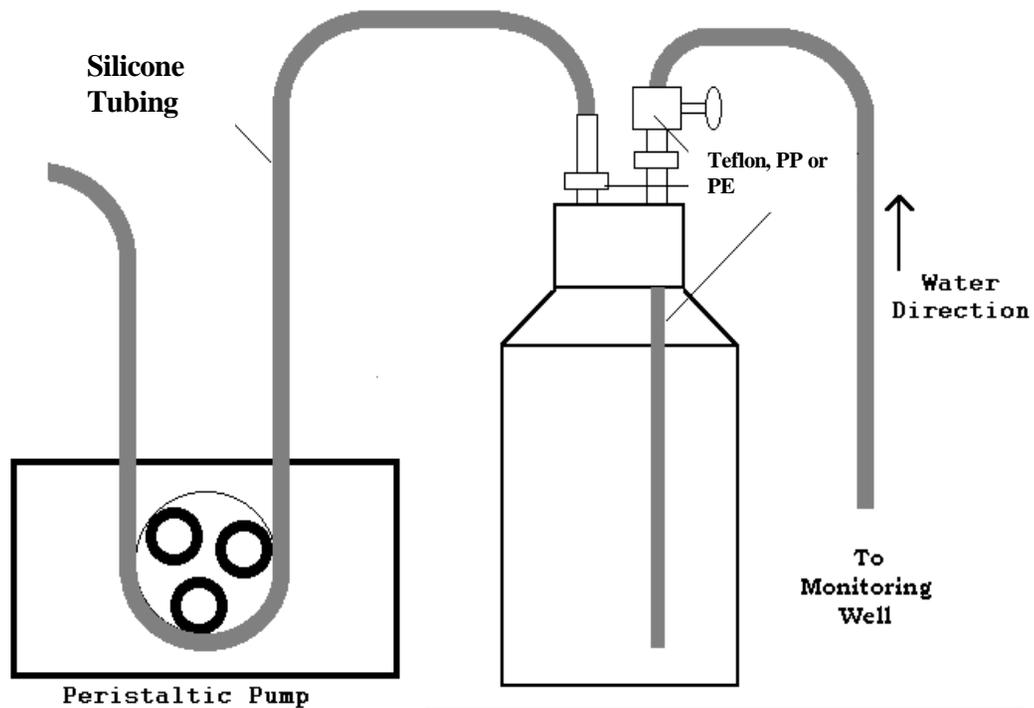
**NA – Not Applicable**

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

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

<sup>1</sup>Daily 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 (HNO<sub>3</sub>) that is suitable for use for metals analysis. Use concentrated HNO<sub>3</sub> or 1:1 HNO<sub>3</sub> 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 HNO<sub>3</sub> or 1:1 HNO<sub>3</sub> 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 Cl<sub>2</sub>.

### 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>
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#### 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 (HNO<sub>3</sub>).

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 HNO<sub>3</sub> or 1:1 HNO<sub>3</sub> 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 2100. SURFACE WATER SAMPLING**

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
- FS 2000 General Aqueous Sampling
- FS 2400 Wastewater Sampling
- FT 1000 – FT 2000 Field Testing and Calibration Procedures

### 1. INTRODUCTION AND SCOPE

1.1. This section presents standard operating procedures to be used to consistently collect representative surface water samples. Each collection event must be performed so that samples are neither contaminated nor altered from improper handling.

1.2. The following topics include acceptable equipment selection and equipment construction materials; and standard grab, depth-specific and depth-composited surface water sampling techniques. Information regarding sample types and flow- or time-weighted aqueous sampling is found in FS 2420.

### 2. GENERAL CAUTIONS

2.1. When using watercraft, take samples near the bow, away and upwind from any gasoline outboard engine. Orient watercraft so that bow is positioned in the upstream direction.

2.2. When wading, collect samples upstream from the body.

2.3. Avoid disturbing sediments in immediate area of sample collection.

2.4. Collect water samples prior to taking sediment samples when obtaining both from the same area (site).

2.5. Consider the representativeness of selected sampling locations, for example, when attempting to characterize a water body that may be stratified or heterogeneous.

2.6. Unless dictated by permit, program or order, sampling at or near structures (e.g., dams, weirs or bridges) may not provide representative data because of unnatural flow patterns.

2.7. Collect surface water samples from downstream towards upstream.

### 3. EQUIPMENT AND SUPPLIES

3.1. Use sampling equipment constructed of materials consistent with the analytes of interest. Refer to FS 1000, Tables 1000-1 and 1000-2 for material selection. Select equipment based on the analytes of interest, the specific equipment use and the available equipment. Refer to FS 1000, Table 1000-3 for selection of appropriate equipment.

- 3.2. For information on sample container size and construction, preservation and holding time requirements, see FS 1000, Tables 1000-4, 1000-5, 1000-8, 1000-9 and 1000-11.
- 3.3. For information on sampling equipment cleaning requirements, see FC 1000.
- 3.4. For information on documentation requirements, see FD 1000.

## FS 2110. **SURFACE WATER SAMPLING TECHNIQUES**

Use the following protocols when collecting surface water samples. Adhere to all general protocols applicable to aqueous sampling detailed in FS 2000 when following the surface water sampling procedures addressed below.

1. **MANUAL SAMPLING:** Use manual sampling for collecting grab samples for immediate in-situ field analyses. Also use manual sampling in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to observe and/or note unusual conditions.

### 1.1. Surface Grab Samples

See FS 2000, section 1.2. for discussions concerning the appropriate uses of grab samples.

Collect surface grab samples within the top 12 inches of the water column. Avoid skimming the surface of the water during collection unless specifically required by the sampling plan. Very shallow water bodies require careful techniques of sample collection to avoid disturbing sediments

Where practical, use the actual sample container as the collection device (direct grab). Sample containers attached to poles are also considered direct grabs.

The use of unpreserved sample containers is encouraged since the same container can be submitted for laboratory analysis after appropriate preservation. This procedure reduces sample handling and potential loss of analytes or contamination of the sample from other sources (e.g., additional sampling equipment, environment, etc.).

#### 1.1.1. Direct Grab Technique

##### 1.1.1.1. Using an unpreserved sample container to collect the sample:

- Remove the container cap and slowly submerge the container, opening first, into the water.
- Invert the bottle so the opening is upright and pointing towards the direction of water flow (if applicable). Allow water to run slowly into the container until filled.
- Return the filled container quickly to the surface.
- Pour out a small volume of sample away from and downstream of the sampling location. This procedure allows for addition of preservatives and sample expansion. Do not use this step for volatile organics or other analytes where headspace is not allowed in the sample container.
- Add preservatives, if required, securely cap container, label and complete field notes.

##### 1.1.1.2. Using a sample container with premeasured preservative to collect the sample. (An unpreserved sample container may also be used with this technique.)

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- Submerge the unopened sample container to the appropriate level.
- Turn the container so that the opening is upright and towards the direction of water flow (if applicable).
- Open the container and allow the water to run into the container almost full (leave an air space).
- Cap the container and return to the surface.

1.1.1.3. If preservatives have been added, invert the container several times to ensure sufficient mixing of sample and preservatives.

1.1.1.4. Check preservation of the sample and adjust pH with additional preservative, if necessary. When a pH adjustment is made and a prepreserved container was used to collect the sample, always check all containers for proper preservation.

1.1.2. Sampling with an Intermediate Vessel or Container: If the sample cannot be collected directly into the sample container to be submitted to the laboratory use an unpreserved sample container or an intermediate vessel (e.g., beakers, buckets or dippers) to obtain the sample. These vessels must be appropriately cleaned and constructed including any poles or extension arms used to access the sample location.

1.1.2.1. Rinse the intermediate vessel with ample amounts of site water prior to collecting the first sample. Discard rinsate away from or downstream of the sampling location.

1.1.2.2. After adequate rinsing, fill the intermediate vessel with sample water. Minimize agitation of the sample.

1.1.2.3. Fill sample containers from the intermediate vessel. Minimize agitation during filling. Do not touch the sample container with the intermediate vessel.

1.1.2.4. Leave adequate headspace in the sample container. This procedure allows for addition of preservatives (if required) and sample expansion. Do not use this step for volatile organics or other analytes where headspace is not allowed in the sample container.

1.1.2.5. Add preservatives if required, securely cap container, label and complete field notes.

1.1.2.6. Invert the container several times to ensure sufficient mixing of sample and preservatives.

1.1.2.7. Check preservation of the sample and adjust pH with additional preservative, if necessary.

1.1.3. Pump and Tubing: Use appropriate pumps, equipment, and tubing. (See restrictions listed in FS 1000 Tables FS 1000-1 through 1000-3).

**Do not collect oil & grease, TRPH or FL-PRO samples with a pump. See FS 2000 for proper collection procedures for extractable organics and volatile organic compounds.**

1.1.3.1. Lower tubing to a depth 6-12 inches below water surface, where possible.

1.1.3.2. Pump several tubing volumes through the system to flush the tubing prior to collecting the first sample.

1.1.3.3. Fill individual sample bottles via the discharge tubing, being careful not to remove the inlet tubing from the water.

1.1.3.4. Do not touch the discharge tubing to the sample container.

1.1.3.5. Leave adequate headspace in the sample container. This procedure allows for addition of preservatives (if required) and sample expansion. Do not use this step for volatile organics or other analytes where headspace is not allowed in the sample container.

1.1.3.6. Add preservatives if required, securely cap container, label and complete field notes.

1.1.3.7. Invert the container several times to ensure sufficient mixing of sample and preservatives.

1.1.3.8. Check preservation of the sample and adjust pH with additional preservative, if necessary.

1.2. Depth Grab Samples: Examples of equipment that may be used for depth grab sampling include Kemmerer, Niskin, Van Dorn and similar samplers; pumps with tubing and double check-valve bailers. See restrictions listed in FS 1000 Tables 1000-1, 1000-2 and 1000-3. Do not collect oil & grease, TRPH or FL-PRO samples with a pump. See FS 2000 for proper collection procedures for extractable organics and volatile organic compounds.

1.2.1. Kemmerer, Niskin and Van Dorn Type Devices

1.2.1.1. Many of these samplers are constructed of plastic and rubber that preclude their use for all volatile and extractable organic sampling. Some newer devices are constructed of stainless steel or are all Teflon or Teflon-coated. These are acceptable for all analyte groups without restriction.

1.2.1.2. Measure the water column to determine maximum depth and sampling depth prior to lowering the sampling device.

1.2.1.3. Mark the line attached to the sampler with depth increments so that the sampling depth can be accurately recorded.

1.2.1.4. Lower the sampler slowly to the appropriate sampling depth, taking care not to disturb the sediments.

1.2.1.5. At the desired depth, send the messenger weight down to trip the closure mechanism.

1.2.1.6. Retrieve the sampler slowly.

1.2.1.7. Rinse the sampling device with ample amounts of site water prior to collecting the first sample. Discard rinsate away from and downstream of the sampling location.

1.2.1.8. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described in sections 1.1.3.3 – 1.1.3.8 above.

1.2.2. Double Check-Valve Bailers: Collect samples using double check-valve bailers if the data requirements do not necessitate a sample from a strictly discrete interval of the water column. Bailers with an upper and lower check-valve can be lowered through the water column and water will continually be displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved.

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1.2.2.1. Sampling with this type of bailer must follow the same protocols outlined in section 1.2.1 above except that a messenger weight is not applicable.

1.2.2.2. Although not designed specifically for this kind of sampling, a bailer is acceptable when a mid-depth sample is required.

1.2.2.3. Note: This sampler does not perform as well as the devices described above or the pump and tubing described in section 1.2.3 below.

1.2.2.4. As the bailer is dropped through the water column, water is displaced through the body of the bailer. The degree of displacement depends upon the check-valve ball movement to allow water to flow freely through the bailer body.

1.2.2.5. Slowly lower the bailer to the appropriate depth. Upon retrieval, the two check-valves seat, preventing water from escaping or entering the bailer.

1.2.2.6. Rinse the sampling device with ample amounts of site water prior to collecting the first sample.

1.2.2.7. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described in sections 1.1.3.3 – 1.1.3.8 above.

1.2.3. Pump and Tubing: Use appropriate pumps, equipment and tubing. (See restrictions listed in FS 1000 Tables 1000-1, 1000-2 and 1000-3). Do not collect oil & grease, TRPH or FL-PRO samples with a pump. See FS 2000 for proper collection procedures for extractable organics and volatile organic compounds.

1.2.3.1. Measure the water column to determine the maximum depth and the sampling depth.

1.2.3.2. Tubing will need to be tied to a stiff pole or be weighted down so the tubing placement will be secure. Do not use a lead or metallic weight if collecting metals samples. Any dense, non-contaminating, non-interfering material will work (brick, stainless steel weight, etc.). Tie the weight with a lanyard (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.

1.2.3.3. Pump several tubing volumes through the system to flush the tubing prior to collecting the first sample.

1.2.3.4. Fill the individual sample bottles via the discharge tube, being careful not to remove the inlet tubing from the water. Do not touch the discharge tubing to the sample container.

1.2.3.5. Leave adequate headspace in the sample container. This procedure allows for addition of preservatives (if required) and sample expansion. Do not use this step for volatile organics or other analytes where headspace is not allowed in the sample container.

1.2.3.6. Add preservatives if required, securely cap container, label and complete field notes.

1.2.3.7. Invert the container several times to ensure sufficient mixing of sample and preservatives.

1.2.3.8. Check preservation of the sample and adjust pH with additional preservative, if necessary.

2. AUTOMATIC SAMPLERS: Use automatic samplers when several sites are to be sampled at frequent intervals or when a continuous sample is required. Composite samplers can be used

to collect time composite or flow proportional samples (see FS 2000, section 1.3 for discussions on types of composite and appropriate use of composite sampling). Use appropriate equipment and tubing. (See restrictions listed in FS 1000 Tables 1000-1, 1000-2 and 1000-3). Do not collect oil & grease, TRPH or FL-PRO samples with automatic samplers unless required by the sampling plan. See FS 2000 for proper collection procedures for extractable organics and volatile organic compounds.

The use of automatic samplers for collecting surface water samples will more frequently run into situations where sampling equipment is deployed on-site for a long term or dedicated to the site.

## 2.1. Installing and Programming the Composite Sampler

2.1.1. Use all new or precleaned pump tubing each time the sampler is brought to the field and set up. If the automatic sampler is deployed in the field for extended periods, it is recommended to replace the tubing at a minimum of every six months. Other replacement schedules may be required, depending on the specific installation and project requirements. Inspect the tubing each time the composite-sample container is picked up. If there is evidence of loss of elasticity or discoloration or other conditions that would impact the quality of the sample (such as algal growth), or the pumping flow rate, then replace the tubing. Select the tubing for the pump head and sampling train according to the analytes of interest and the allowable construction materials specified in FS 1000 Table FS 1000-1, 1000-2 and 1000-3.

2.1.1.1. Cut the proper length of precleaned Teflon or Tygon tubing.

2.1.1.2. Equipment Blanks: Collect equipment blanks each time the tubing is changed or at a frequency of 5% of the tubing changes, whichever is less. Collect a minimum of one blank each year. Collect the blank by passing analyte-free water through the equipment that is exposed to the sample.

- Composite sample containers may be cleaned either in the field or in a fixed base operation. Demonstrate cleaning effectiveness by collecting equipment blanks on the composite sample containers according to the frequency specified in FQ 1000. Collect sample container equipment blanks by adding analyte-free water to the cleaned sample container, mix the water thoroughly within the container and then pour off an aliquot for analysis.

2.1.1.3. Put the collection sieve and tubing in the appropriate sample location, using conduit if necessary to hold it in place. Ensure the supporting conduit does not contaminate the incoming sample water.

2.1.1.4. Program the sampler per manufacturer's directions and as required in the permit or work plan conditions.

2.1.1.5. Automatic Sampler Security: Place a lock or seal on the sampler to prevent or detect tampering. This procedure, however, does not prevent tampering with the sampler tubing. See additional discussions on sample security in FS 2410, section 2.3.2.

## 2.2. Sample Acquisition

2.3.1. At the end of each sampling period, stir the contents of the composite jug and transfer the contents into the respective containers. If the sampler was configured to collect discrete samples ensure that the contents of each container are adequately mixed while pouring the sample into the sample container.

2.3.2. Immediately preserve the sample, if required, securely cap container, label and complete field notes.

2.3. Long Term Deployment of Automatic Composite Samplers: In certain sampling situations, automatic composite samplers are permanently installed at surface water stations and remain in the field for months or even years. Under these conditions, there are specific sampling issues that need to be addressed.

2.3.1. Sample Preservation

2.3.1.1. If the only analyte of interest is Total Phosphorus and the project is unrelated to an NPDES permit, the sample must be chemically preserved with sulfuric acid ( $H_2SO_4$ ) but it need not be cooled to 4°C with wet ice.

- The acid must be in the container prior to drawing the first composite sample into the container.

When using large (i.e., 3 gallon) composite sample containers, and there is potential for the sample size to vary greatly due to variable flow rates at the site, the volume of acid for preservation should be small (e.g., 1 to 2 mL of 50%  $H_2SO_4$ ). **Do not over acidify the sample.** Upon sample pick-up, if needed, add additional acid to achieve the proper pH adjustment for preservation.

- If parameters other than total phosphorus are to be analyzed, appropriate additional preservation (e.g., cooling with ice or refrigeration) is required.

2.3.1.2. Deviations from these SOPs concerning preservation and holding times relating to remote and long term deployments due to site specific considerations must be agreed upon by project management.

2.3.2. Cleaning Requirements

2.3.2.1. Clean composite sampler containers after collection of each composite sample using cleaning solutions and procedures specified in FC 1140, sections 5 through 9.

2.3.2.2. Composite sample containers may be cleaned either in the field or in a fixed based operation. Demonstrate cleaning effectiveness by collecting equipment blanks on the composite sample containers according to the frequency specified in FQ 1000. Collect sampler container equipment blanks by adding analyte-free water to the cleaned sample container, mix the water thoroughly within the container and then pour off an aliquot for analysis.

2.3.2.3. Inspect and replace tubing at a minimum of every six months or when applicable, as discussed in section 2.1.1 above. Collect equipment blanks as specified in section 2.1.1.2 above. If the tubing is being replaced for multiple autosamplers at the same time, one equipment blank may be collected on the entire length of replacement tubing. Collect this equipment blank by passing analyte-free water through the entire length of new tubing.

## 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:  $\pm 0.2$  Standard Units
- Specific Conductance:  $\pm 5.0\%$  of reading
- Dissolved Oxygen:  $\leq 20\%$  Saturation
- Turbidity:  $\leq 20$  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  $\leq 20$  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  $\leq 20$  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:  $\pm 0.2$  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  $\leq 500$  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  $\leq 100$  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  $\leq 500$  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  $\leq 100$  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  $\leq 100$  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  $\leq 1$  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  $\leq 100$  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  $\leq 100$  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  $\leq 100$  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  $\leq 100$  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- $\mu$ 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**

<b>Activity</b>	<b>Equipment Type</b>
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

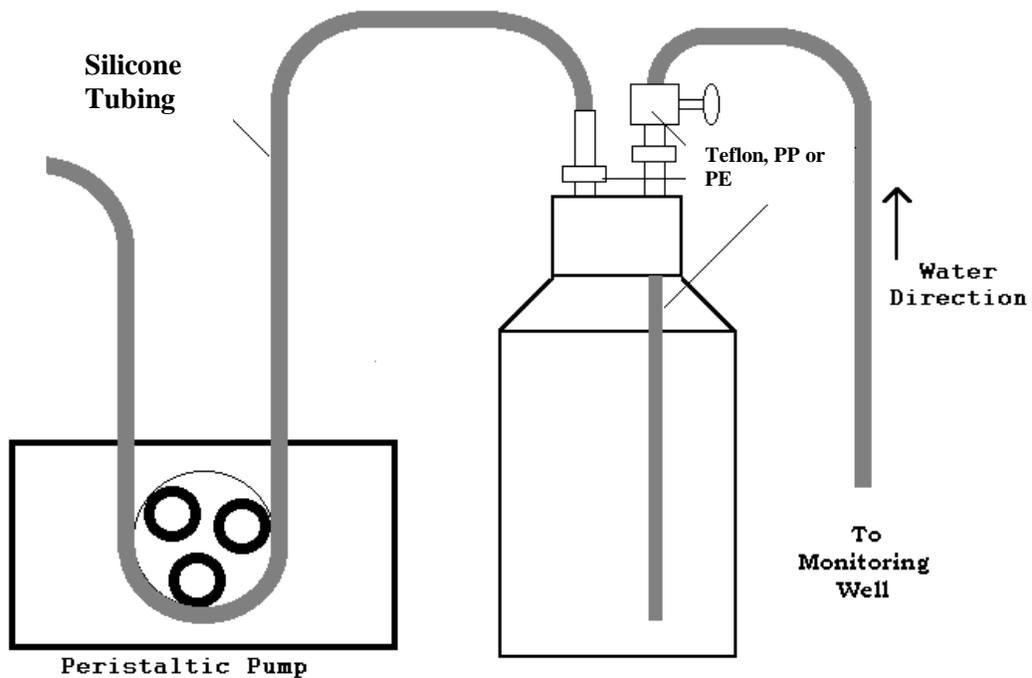
**Table FS 2200-3  
 Allowable Uses for Bailers**

• 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  $\leq 10$  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 bailer<sup>1</sup> is placed above the screen at the top of the water column.*

<sup>1</sup> 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 bailer<sup>1</sup> 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  $\geq 2$  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  $\geq 2$  to 3 minutes apart.

Purging Completion

If Dissolved Oxygen is  $\leq 20\%$  of saturation for the measured temperature and Turbidity is  $\leq 20$  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  $\pm 0.2$  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  $\pm 0.2$  Standard Units  
Specific Conductance  $\pm 5.0\%$  of reading  
Dissolved Oxygen  $\pm 0.2$  mg/L or readings are within 10% (whichever is greater).  
Turbidity  $\pm 5$  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  $\leq 100$  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  $-10^{\circ}\text{C}$  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.

After collecting the samples, remove the temporary casing (if used) and fill the hole filled with the excavated soil.

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.

<b>Table FT 1000-1: Field Testing Acceptance Criteria</b>	
<b>Parameter</b>	<b>Acceptance Criteria</b>
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

<b>Table FT 1000-1: Field Testing Acceptance Criteria</b>	
<b>Parameter</b>	<b>Acceptance Criteria</b>
<b>pH (FT 1100)</b>	<b>± 0.2 Standard pH Units of buffer or more stringent program criteria</b>
<b>Specific Conductance (FT 1200)</b>	<b>± 5% of standard value</b>
<b>Temperature (FT 1400)</b>	<b>± 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values</b>
<b>Dissolved Oxygen (FT 1500)</b>	<b>± 0.3 mg/L of theoretical value (see Table FT 1500-1)</b>
<b>Turbidity (FT 1600)</b>	<b>0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value &gt; 100 NTU: ± 5% of standard value</b>
<b>Total Residual Chlorine (FT 2000)</b>	<b>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</b>

## 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<sup>+</sup>-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  $\pm 0.2$  pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range. Certain regulatory programs may have more stringent acceptance criteria.
    - 2.3.2.4. After sample measurement(s), perform a continuing calibration verification (CCV). Read a buffer as a sample. To be acceptable, a

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calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

- 2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.
- 2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.
- 2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).
  - 2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.
  - 2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.
  - 2.6.3. Wait for stabilization, and read the pH value.
  - 2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.
3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.
4. DOCUMENTATION
  - 4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.
    - 4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.
      - 4.1.1.1. Document acceptable verification of any standard used after its expiration date.
    - 4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.
      - 4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.
      - 4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.
    - 4.1.3. Record the grade of standard or reagent used.
    - 4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.
      - 4.1.4.1. Record the date of preparation for all in-house formulations.
    - 4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).
  - 4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

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- 4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.
  - 4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.
    - 4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
  - 4.2.3. Record the time and date of all initial calibrations and all calibration verifications.
  - 4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
  - 4.2.5. Record the name of the analyst(s) performing the calibration.
  - 4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
    - Type of standard or standard name (e.g., pH buffer)
    - Value of standard, including correct units (e.g., pH = 7.0 SU)
    - Link to information recorded according to section 4.1 above
  - 4.2.7. Retain manufacturers' instrument specifications.
  - 4.2.8. Document whether successful initial calibration occurred.
  - 4.2.9. Document whether each calibration verification passed or failed.
  - 4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
    - 4.2.10.1. Document date and time of any corrective action.
    - 4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
  - 4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 4.3. Record all field-testing measurement data, to include the following:
- Project name
  - Date and time of measurement or test (including time zone, if applicable)
  - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
  - Latitude and longitude of sampling source location (if required)
  - Analyte or parameter measured
  - Measurement or test sample value
  - Reporting units
  - Initials or name of analyst performing the measurement
  - Unique identification of the specific instrument unit(s) used for the test(s)

## **FT 1200. Field Measurement of Specific Conductance (Conductivity)**

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

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

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

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

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

### 3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100  $\mu\text{mhos/cm}$  or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100  $\mu\text{mhos/cm}$  standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100  $\mu\text{mhos/cm}$ .

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100  $\mu\text{mhos/cm}$ , only one standard at 100  $\mu\text{mhos/cm}$  standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100  $\mu\text{mhos/cm}$  standard is 95 to 105  $\mu\text{mhos/cm}$ . If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using

the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}$$

Where: K = conductivity in  $\mu\text{mhos/cm}$  at 25°C

$K_m$  = measured conductivity in  $\mu\text{mhos/cm}$  at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}$$

Refer to SM2510B, 20<sup>th</sup> edition, if other calculations (i.e., determining cell constant, etc.) are required. See FT 1210 below.

3.5 *In situ* Measurements at Depth or With Flow-through Cells: After calibrating the instrument as outlined in 3.2 above, follow the manufacturer's instructions to measure the conductivity of the sample.

3.5.1. For *in situ* measurements immerse the probe at the desired depth and wait for stabilization of the reading and record its value. Follow a similar procedure when using a flow-through cell.

3.5.1.1 Preferably measure groundwater sample conductivity *in situ* with a downhole probe or in a flow-through system.

4. PREVENTATIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

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- 5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
- 5.2.3. Record the time and date of all initial calibrations and all calibration verifications.
- 5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
- 5.2.5. Record the name of the analyst(s) performing the calibration.
- 5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
  - Type of standard or standard name (e.g., conductivity standard)
  - Value of standard, including correct units (e.g., conductivity = 100 µmhos/cm)
  - Link to information recorded according to section 5.1 above
- 5.2.7. Retain manufacturers' instrument specifications.
- 5.2.8. Document whether successful initial calibration occurred.
- 5.2.9. Document whether each calibration verification passed or failed.
- 5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
  - 5.2.10.1. Document date and time of any corrective action.
  - 5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 5.3. Record all field-testing measurement data, to include the following:
  - Project name
  - Date and time of measurement or test (including time zone, if applicable)
  - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
  - Latitude and longitude of sampling source location (if required)
  - Analyte or parameter measured
  - Measurement or test sample value
  - Reporting units
  - Initials or name of analyst performing the measurement
  - Unique identification of the specific instrument unit(s) used for the test(s)

## FT 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 µmho/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 (D<sub>0</sub>) is given by:

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

Where T and T<sub>0</sub> 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 - D<sub>0</sub>) 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 H<sub>2</sub>S) 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

<b>Table FT 1500-1: Solubility of Oxygen in Water</b>			
<b>at Atmospheric Pressure<sup>1,2</sup></b>			
<b>Temperature</b>	<b>Oxygen Solubility</b>	<b>Temperature</b>	<b>Oxygen Solubility</b>
<b>°C</b>	<b>mg/L</b>	<b>°C</b>	<b>mg/L</b>
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.

- 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)

**APPENDIX B**

**ANALYTICAL LABORATORY  
STANDARD OPERATING PROCEDURES**

## **EMPIRICAL LABORATORY**

## 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	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Di-isopropyl ether
GC/MS	8260B	ETBE
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	t-Butyl alcohol
GC/MS	8260B	tert-Amyl methyl ether
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	3,5-Dinitroaniline
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	8011	1,2-Dibromoethane (EDB)
GC/ECD	8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7470A	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
IC	300.0	Chloride
IC	300.0	Fluoride

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
IC	300.0	Nitrate
IC	300.0	Nitrite
IC	300.0	Sulfate
IC	9056A	Chloride
IC	9056A	Fluoride
IC	9056A	Nitrate
IC	9056A	Nitrite
IC	9056A	Sulfate
Titration	SM 2320B 20th ed.	Alkalinity
ISE	SM 4500 B, D, 20th ed.	Ammonia
UV/Vis	7196A	Hexavalent Chromium
Colorimetric	353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th edition	Total Phosphorus
UV/Vis	SM 4500 PE, 20th edition	Ortho-Phosphorus
TOC	9060A/SM5310C, 20 <sup>th</sup> edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th edition	TDS
Colorimetric	9012A/B	Cyanide
Physical	1010A	Ignitability
Physical	9095B	Paint Filter
Probe	9040B/C	pH
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Preparation	1311	TCLP
Preparation	3005A	Metals digestion
Preparation	3010A	Metals digestion
Preparation	3510C	Organics Liquid Extraction
Preparation	5030A/B	Purge and Trap Water

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBd)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7471A/B	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
UV/Vis	7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	9034	Sulfide
Probe	9045D	pH
Preparation	Method	Type
Preparation	1311	TCLP
Preparation	1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	3050B	Metals Digestion
Preparation	3546	Organics Microwave Extraction
Preparation	3541	Organics Soxhlet Extraction
Preparation	3550B	Organics Sonication
Preparation	SM 2540B 20th edition	Percent Solids (Percent Moisture)
Preparation	5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: \_\_\_\_\_



R. Douglas Leonard  
Chief Technical Officer

Date: November 30, 2009

Issued: 11/30/09

**METALS DIGESTION/PREPARATION**

**METHODS**

**USEPA SW846**

**3005A, 3010A, 3030C, 3031, 3050B**

**USEPA CLPILM 04.1 Aqueous & Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June, 2003)**

**Addendum for USEPA CLPILM 05.2 Aqueous & Soil/Sediment**

**USEPA Methods for Chemical Analysis of Water and Wastes**

**200.7, Standard Methods 3030C**

**SOP NUMBER:**

**SOP-100**

**REVISION NUMBER:**

**19**

**APPROVED BY:**

*Betty DeVill*

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**QUALITY ASSURANCE OFFICER**

**07/25/06**

**EFFECTIVE DATE**

**04/20/09**

**DATE OF LAST REVIEW**

## METALS DIGESTION/PREPARATION

### References:

**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3031, 3050B**

**USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C**

**See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

## I. SCOPE AND APPLICATION

### A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
  - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
  - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
  - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
  - a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

**B. SOLIDS**

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
  - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
  - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
  - a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

**C. OILS**

1. Method 3031, "Digestion Procedure for Oils".
  - a. This method is used to prepare samples containing oils, greases or waxes for analysis by inductively coupled argon plasma emission spectroscopy (ICP).

**D. NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

**II. SUMMARY OF METHODS**

- A. A representative sample of water, soil or oil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration must be used in order to reach that lower level. Care

must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike ( not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

### **III. SAMPLE HANDLING AND PRESERVATION**

#### **A. AQUEOUS**

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO<sub>3</sub> to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO<sub>3</sub> is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

#### **B. SOLIDS**

1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

#### **C. OILS**

1. Samples are taken in high density polyethylene bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

### **IV. INTERFERENCES**

#### **A. AQUEOUS**

1. Method 3005A and USEPA CLPILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy", SW846, July, 1992.
  - a. This digestion procedure may not be sufficiently vigorous to destroy some metal complexes.
2. Method 200.7

3. Method 3010A
  - a. See method 6010B.

## B. SOLIDS

1. Method 3050B
  - a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.
2. USEPA CLP ILM0 4.1
  - a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

## C. OILS

1. Method 3031
  - a. These digestates can have very high dissolved solids, which may necessitate the use of internal standards, dilutions, or the method of standard addition.

## V. SAFETY

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
- B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
- C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

## VI. EQUIPMENT/APPARATUS

- A. Fume hood, Labconco or equivalent.

- B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.
- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
- D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
- E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
- F. Beckman CS-6R centrifuge.
- G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
- H. Whatman No. 41 filter paper or equivalent.
- I. Whatman No. 42 filter paper or equivalent.
- J. Whatman 0.45 micron filter paper or equivalent.
- K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
- L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
- M. Manual Sample Mill
- N. Wiley Sample Mill
- O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

## **VII. REAGENTS AND STANDARD PREPARATION**

### **A. REAGENTS**

1. Metals grade Nitric acid ( $\text{HNO}_3$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid ( $\text{HCl}$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid ( $\text{H}_2\text{SO}_4$ ). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
9. Base oil, analyte-free. Oil should be analyzed to determine level of impurities. If method blank is < MDL, then the reagent can be used.

## **B. STANDARDS**

### **1. Traceability**

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

## 2. PREPARATION

### A. Laboratory control sample

#### 1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO<sub>3</sub>, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIIM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO<sub>3</sub>, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO<sub>3</sub> and 5% HCl. Use 0.125 mLs HNO<sub>3</sub> and 0.3125 mLs HCl to each 50 mL vessel.

## 2. Solids

a. A 1.0  $\pm$ 0.02 gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and recorded in the sample digestion logbook.

## 3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

## B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

## VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 85° for oil samples. Record in digestion logbook.

## IX. PROCEDURE

### A. Glassware preparation for oil digestion or when the hot-block can not be used:

1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
2. Rinse glassware with reagent water that contains 5% HNO<sub>3</sub> and 5% HCl followed by a rinse with reagent water.
3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

### B. Aqueous sample filtration (for dissolved metals):

1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO<sub>3</sub> followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
2. Rinse a 0.45 micron filter with 1:5 HNO<sub>3</sub> thoroughly, followed by D.I. water.
3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
4. Discard the first 50 to 100 mL.
5. A preparation blank must be taken through the filtration step and analyzed with the sample.
6. Preserve the sample with HNO<sub>3</sub> to pH<2.
7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO<sub>3</sub>. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

### C. Aqueous sample preparation

1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".
  - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
  - b. Add 0.50 mL ( 1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample.
  - c. Add 2.5 mL ( 5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
  - d. Cover the sample with a ribbed watch glass or equivalent source.
  - e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, ( 12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
  - f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - g. Bring sample to its predigestion volume ( or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.
  - h. The sample is now ready for analysis.
  - i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
- 2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
  - b. Add 1.0 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Add 2.50 mL concentrated HCl to the sample.
  - d. Cover the sample with a ribbed watch glass or equivalent source.
  - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
  - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
  - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
  - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - i. The sample is now ready for analysis.
  - j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel.
  - b. Add 1.5 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Cover the sample with a ribbed watch glass.
  - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature

being recorded in the log book. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.

- e. Add another 1.5 mL portion of concentrated HNO<sub>3</sub> to the sample.
- f. Cover the sample with a ribbed watch glass.
- g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
- h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
- i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
- j. Cover the digestion vessel and reflux for an additional 15 minutes.
- k. Bring sample to its predigestion volume in digestion vessel.
- l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

**Note:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

- m. The sample is now ready for analysis.
  - n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
- b. Add 2.5 mL 1:1 HCl to the sample.
- c. Heat 15 minutes in a hot bath.
- d. Filter through a membrane filter.
- e. Adjust filtrate volume to 50 mL with DI water.
- f. Transfer to ICP analyst.

#### D. Solid sample preparation

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

#### **Grinding of Vegetation Samples**

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill

or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"

- a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
- b. Add 10 mL of 1:1 nitric acid ( $\text{HNO}_3$ ), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated  $\text{HNO}_3$ , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
- c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
- d. Continue to add 30%  $\text{H}_2\text{O}_2$  in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30%  $\text{H}_2\text{O}_2$ .)
- e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated  $\text{HNO}_3$ , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v)  $\text{HNO}_3$ . The sample is now ready for analysis.

- f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, “**Acid digestion of Sediments, Sludges and Soils**”

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

**NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.**

- c. Add 5 mL D.I. water and 5 mL concentrated  $\text{HNO}_3(1:1)$ , mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at  $95^\circ\text{C}$  for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book
- d. Allow the sample to cool. Add 5 mL concentrated  $\text{HNO}_3$ , replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by  $\text{HNO}_3$ , repeat this step (addition of 5 mL of concentrated  $\text{HNO}_3$ ) over and over until no brown fumes are given off by the sample indicating the complete reaction with  $\text{HNO}_3$ . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at  $95^\circ\text{C} \pm 5^\circ\text{C}$  for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of  $\text{H}_2\text{O}_2$  if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H<sub>2</sub>O<sub>2</sub> to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H<sub>2</sub>O<sub>2</sub>.)

- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
  - g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
  - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.
- NOTE1:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
- NOTE2:** To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.
- j. The sample is now ready for analysis.
  - k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## E. Oils

## 1. Method 3031, "Digestion Procedure for Oils"

**NOTE: THIS METHOD IS VERY TIME CONSUMING--  
DISCUSS SUB-CONTRACTING SAMPLES WITH  
YOUR SUPERVISOR AS SOON AS THEY COME IN  
THE DOOR.**

- a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.
  
- g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to potassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

- h. Cautiously add 1.0 mL concentrated  $\text{H}_2\text{SO}_4$ , and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The  $\text{H}_2\text{SO}_4$  can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

NOTE: To prevent a strong exothermic reaction,  $\text{H}_2\text{SO}_4$  should be added dropwise to all samples unfamiliar to the analyst and to all samples that are known to be highly reactive.

The reaction can take several seconds to begin, but when it occurs it will be very quick, vigorous, and exothermic. Generally larger sample sizes will react faster than smaller. Likewise, lower average molecular weight materials will react faster than heavier. Do not be misled by an initial lack of reactivity. A grey-white vapor will be ejected from the beaker ( $\text{SO}_3$ ) and splattering and bubbling can occur. The beaker will become very hot. This step is complete when no more gases are given off and the sample would be a thick black lumpy paste. Allow the beaker to cool as needed.

NOTE: Care must be taken when working with very light organic materials, such as diesel fuels, as they may flash. Generally, the lower the average molecular weight of the material correlates to a greater danger of flashing. The danger of flashing is reduced by adding the sulfuric acid dropwise.

NOTE: If more than 10% of the sample is aromatic material, such as xylene, only a little grey-white vapor will form. This will reduce accuracy and complicate nebulization. If there is a significant amount of non-hydrocarbon material, a sputtering reaction will occur and black  $\text{MnO}_2$  particulates will be given off. See section (b.) above under procedure.

- i. Add 2 mL of concentrated  $\text{HNO}_3$  and stir. This reaction will be slightly exothermic. If larger sample sizes are used, it is not always necessary to increase the volume of  $\text{HNO}_3$  proportionately, depending on analytical needs. Some reddish-brown vapor ( $\text{NO}_2$ ) may be given off. Allow the reaction to continue until complete, that is when the digestate no longer gives off fumes. Allow the beaker to cool as needed.
- j. Add 10 mL of concentrated  $\text{HCl}$  and stir. If larger sample sizes are used, it is not always necessary to increase the volume of  $\text{HCl}$  proportionately, depending on analytical needs. This reaction will be slightly exothermic and gas formation and foaming will occur. Lighter oils will foam more than will heavier oils. If excess foaming occurs, add water to prevent sample loss. Allow the beaker to cool as needed.
- k. Heat the beaker until there is no further gas evolution. (temperature should not exceed  $150\text{ }^\circ\text{C}$  to prevent volatilization). There may be additional foaming or other milder reactions which may result in overflow from the beaker. If excess foaming occurs, either remove the beaker from the heating source until foaming subsides or add

sufficient water to prevent overflow. The final digestate should be a clear yellow liquid with black or dark reddish-brown particulates.

- l. Filter the digestate through Whatman 41 filter paper and collect filtrate in a volumetric flask or beaker.
- m. Wash the digestion beaker and filter paper, while still in the funnel, with no more than 5 mL of hot HCl.

NOTE: The purpose of this next step is to recover antimony, barium, and silver that may not have been completely solubilized. If the sample is not being prepared for these analytes, the next step may be skipped.

- n. (Optional) After having washed the filter paper, remove the filter and residue from the funnel and place it back in the beaker. Add 5 mL of conc. HCl and place the beaker back on the heating source until the filter paper dissolves (temperature should not exceed  $150\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  to prevent volatilization). Remove the beaker from the heating source and wash the cover and sides with reagent grade water and then filter the residue and collect the filtrate in the same flask or beaker as in sections f. and g. above. Allow the filtrate to cool and quantitatively transfer to a volumetric flask. Bring to volume.
- o. (Optional) If the filtrate is collected in a beaker, the filtrate can be heated again to drive off excess HCl. This can reduce matrix effects in sample introduction (temperature should not exceed  $150\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  to prevent volatilization). When sufficient HCl has been removed, remove the beaker from the heating source, allow to cool, and then transfer the contents to a volumetric flask and bring to volume. However, if too much HCl is removed, barium, silver and antimony can be lost.
- p. Analyze the filtrate by ICP-AES. Depending on the final volume selected, the total solids in the digestate may be high enough to cause nebulization problems. Problems due to high dissolved solids may be corrected by 1) following optional Section i., 2) using internal standards, 3) using flow injection analysis, or 4) using other matrix correction procedures.

#### Manganese Removal Steps

NOTE: The purpose of these next steps is to remove the manganese in the digest by precipitating it as manganese ammonium phosphate

under alkaline conditions. Elements that do not form insoluble phosphates, such as arsenic, are filtered out and can be analyzed at lower concentrations.

- q. Take the digestate, or portion of digestate and reduce the volume to remove as much HCl as possible without going below 10 mL. Then add conc.  $\text{NH}_4\text{OH}$  until pH is 7 or greater. For most matrices, the digestate will change colors (often from yellow to brown) at pH 7. A mild exothermic reaction will occur immediately.
- r. Add at least 2 g ammonium phosphate for each 1 g of potassium permanganate used in the digestion and stir. An excess of phosphate is needed for good analyte recovery. Then add enough water and mix to ensure maximum precipitation. A pink or yellow silky amorphous precipitate, manganese ammonium phosphate, will form. If too much  $\text{NH}_4\text{OH}$  is used some of the manganese ammonium phosphate can be solubilized. Stir until precipitation is complete. Some ammonium phosphate may remain unreacted at the bottom of the beaker.
- s. Filter the digestate through Whatman 41 filter paper (or equivalent) and collect filtrate in a volumetric flask or beaker.
- t. Heat the filtrate to volatilize the ammonia (temperature should not exceed  $150\text{ }^\circ\text{C} \pm 5\text{ }^\circ\text{C}$  to prevent volatilization). The volume of filtrate can be reduced by heating to no less than 10 mL. If too much water is removed ammonium chloride formed will solidify. If this occurs, either add enough water to dissolve the solids or filter out the solids and wash the residue with deionized water. The filtrate can be analyzed by ICP-AES.
- u. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## X. CALCULATIONS

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

## XI. QUALITY CONTROL

- A. Digestion

1. Temperature blank
  - a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
  - b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
  
2. Blanks
  - a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples. **NOTE: The blank for OILs MUST include an analyte-free oil or explosive reactions can occur.**
  - b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
  - c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
  - d. Sample is given a unique identifier in the digestion log.
  
3. Laboratory Control Samples
  - a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
  - b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
  - c. For soil/sediment samples, a soil matrix standard reference material (SRM ) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
  - d. Sample is given a unique identifier in the digestion log.
  - e. Recoveries of standard reference materials or laboratory control samples spiked with organo-metallic standards recoveries should be **±25% of their true values for OILS.**

## 4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

**NOTE:** Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

## 5. Blank Spike

- a. This is required for certain projects.

## B. Sample Matrix

**NOTE:** Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

## 1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

**NOTE:** For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium  
Magnesium  
Sodium  
Potassium

- v. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)

**d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

## XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
  - 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
  - 1. Redigest another sample aliquot.
  - 2. Glass beaker dry for an extended period of time? Discard beaker.

## XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
  - 1. All metals glassware must be kept separate from all other laboratory glassware.
  - 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
  - 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
  
- F. Please consult Waste Disposal SOP-405, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### **Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT**

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

**DEFINITIONS** – Refer to SOP-431 for common environmental laboratory definitions.

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**METALS: SOP 103      REVISION #: 18      EFFECTIVE DATE: 041110**

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**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: \_\_\_/\_\_\_/\_\_\_

Data Quality Manager: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Section Supervisor: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

## **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.

## 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

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

<b>Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>					
<b>Mercury by EPA 245.1, 7470A, SOW 4.1 &amp; 5.2</b>	<b>AQUEOUS MDL/DL (ug/L)</b>	<b>AQUEOUS LOD (ug/L)</b>	<b>AQUEOUS ERL/LOQ (ug/L)</b>	<b>AQUEOUS CRQL ILMO 4.1 (ug/L)</b>	<b>AQUEOUS CRQL ILMO 5.2 (ug/L)</b>
<b>Mercury</b>	0.080	0.16	0.20	0.20	0.20

#### Wavelength Table

<b>ANALYTE</b>	<b>WAVELENGTH</b>
<b>Mercury</b>	<b>253.7</b>

### 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 HNO<sub>3</sub>. This solution is recorded in Element and given a unique Element identifier.

## 10.2.2 Calibration Standards

Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

10.2.2.1 Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.)

<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 30 mLs</u>
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

10.2.2.2 Appropriate reagents are added as below in the sample preparation section.

10.2.2.3 Prepare one vessel for each.

10.2.2.4 It is necessary to digest the calibration standards.

## 10.2.3 Calibration Verification Standards

10.2.3.1. Initial calibration verification ( ICV ) solution – 4.0 ug/L

10.2.3.1.1 Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

10.2.3.1.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.1.3 It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.3.2 Continuing calibration verification ( CCV ) solution

10.2.3.2.1 Prepared from the primary source standard.

10.2.3.2.2 Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

10.2.3.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.3.2.4 It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.

#### 10.2.4 Digestion standards

##### 10.2.4.1 Blank Spike

10.2.4.1.1 Prepared from the secondary source standard.

10.2.4.1.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 This solution should be given a unique identifier within Element.

##### 10.2.1.2 Matrix Spikes

10.2.1.2.1 Prepared from the secondary source working standard.

10.2.1.2.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

10.2.1.2.3 Appropriate reagents are added as below in the sample preparation section.

## 11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.

11.2 The holding time for the mercury digestion is 28 days from time of sampling.

## 12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BS’s are analyzed at 0.10ug/L. See [Table 2](#) for acceptance criteria.

12.3 **(Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)**

12.4 Daily

- 12.4.1. **The instrument must be calibrated daily for all projects.**
- 12.4.2 Begin each analysis with an ICV(QCS) second source. The control limits are  $\pm 10\%$  and IPC (CCV) for 245.1, limits are  $\pm 5\%$  and subsequent analyses are  $\pm 10\%$ .
- 12.4.3 Analyze ICB. Control limits ( $<\pm$ MDL for USACE or  $\pm$ RL/CRDL for others and CLP)., depending on method. **No analyte detected  $>2$ 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 ( $<\pm$ MDL for USACE or  $\pm$ RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected  $>2$ 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 ( $<1/2$   $\pm$ RL or  $\pm$ RL/CRDL for common contaminants (DOD) and  $\pm$ RL/CRDL for others and CLP).
    - 12.6.2.1 Employ a minimum of one preparation blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be re-digested and re-analyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.
  - 12.6.3 Laboratory control sample ( BS )
    - 12.6.3.1. Employ a minimum of one laboratory control sample ( BS ) per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are  $\pm 20\%$  for SW846-7470 and  $\pm 15\%$  for 245.1. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.
- 12.7 Sample matrix:

- 12.7.1 Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. CLP does not allow this. Project specific requirements will take precedence in these situations.
- 12.7.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within  $\pm 25\%$  of the true value ( **$\pm 20\%$  for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.  
**NOTE:** For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.
- 12.7.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.
- 12.7.4 For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be  $\pm 10\%$ . The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.
- 12.7.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

## 13.0 Calibration and Standardization

Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1 Set up the instrument with proper operating parameters.

13.1.1 Perkin Elmer Flow Injection Mercury System (FIMS).

13.1.1.1. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. (**Perkin-Elmer tygon tubing, waste**

**and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)**

- 13.1.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
- 13.1.1.3 Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
- 13.1.1.4 Turn on computer and go to icon "AA Win LAB Analyst".
- 13.1.1.5 Go to method; select "Hg CAL 2" then OK.
- 13.1.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.
- 13.1.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.
- 13.1.1.8 Save entered sample list under "Save ....sample 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 SnCl<sub>2</sub>. (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  $\geq 0.995$  ( 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  $\text{KMnO}_4$  the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

**NOTE:** The same amount of  $\text{KMnO}_4$  added to the samples should be present in the standards and blanks.

14.4.5 Add 2.4 mLs of potassium persulfate to each vessel and mix. Cover.

14.4.6 Heat for 2 hours in the block digester at  $95 \pm 2^\circ\text{C}$  (the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool.

14.4.7 Samples may be saved at this point if there is not time to run the whole set that day.

**NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.**

14.5 Sample analysis

14.5.1 Set up the instrument as described in the calibration section above.

14.5.2 When ready to run samples, add 1.8 mLs of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of  $\pm 10\%$  for SW846-7470 and  $\pm 5\%$  for 245.1. Followed by the ICB ( $< \pm \text{MDL}$  for USACE or  $\pm \text{RL/CRDL}$  for others and CLP).

14.5.3 Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of  $\pm 20\%$  for SW846-7470 and  $\pm 10\%$  for 245.1.

14.5.4 CCB must always follow the CCV. Control limits are ( $< \pm \text{MDL}$  for USACE or  $\pm \text{RL/CRDL}$  for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected  $> 2 \times \text{MDL}$  for DOD.**

14.5.5 The auto-sampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ-ICV
10	SEQ-ICB
11	BS

AS LOC	Sample ID
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB

23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

#### 14.6 Data Reporting

14.6.1 Reduce data to result which will be reported.

14.6.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

### 15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Apply a least squares fit to the calibration standards plotting  $\mu\text{g Hg/L}$  versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume ( the 0.1  $\mu\text{g Hg}$  standard will be input as 1.0  $\mu\text{g Hg/L}$  ) ( 0.1 $\mu\text{g Hg}$  / 0.030 L solution ).

15.3 Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.

15.4 Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.

15.5 Report the data as  $\mu\text{g Hg/L}$  of sample.

### 16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.3 mL of the second source standard to 30 mLs with reagent water in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

## 17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

## 19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

### 19.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

19.2.1 ICV (QCS for 245.1)- second source not within  $\pm 10\%$ .

- A. If the problem is with the solution, re-prepare, obtain new stock if necessary.
- B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within  $\pm 20\%$  for SW846 and  $\pm 10\%$  for (245.1,  $\pm 5\%$  for initial IPC and  $+ 10\%$  for subsequent IPCs)

- A. If the problem is with the solution, re-prepare, obtain new stock if necessary.
- B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.
  1. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
  2. If the CCV was biased low, the previous ten samples must be reanalyzed.

### 19.3 CORRECTIVE ACTION: DIGESTION RELATED

19.3.1 The preparation blank less than  $<1/2$  RL or  $\pm$ RL/CRDL for common contaminants (DOD) and  $\pm$ RL/CRDL for others and CLP.

- A. If the problem is with the instrument or stannous chloride.  
Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, re-prepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.
- B. If the problem is with the digestion.  
All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be re-digested/re-analyzed or reported as qualified. The project manager or QA manager will make this determination.
- C. LCS not within control limits (or  $\pm 20\%$ ,  $\pm 15\%$  for **245.1** ).
  - 1. If the problem is with the instrument, reanalyze when instrument is in control if further sample bottles are available.
  - 2. Is the problem is with the digestion.
    - a. If biased low, associated samples must be re-digested.
    - b. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

#### 19.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

19.4.1 Replicate analysis RPD not within  $\pm 20\%$

The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within  $\pm 25\%$  ( **$\pm 20\%$  for DOD projects**)

- A. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
- B. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

19.4.3 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

## 20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage

during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **21.0 References**

- 21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 7470A.*
- 21.2 *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B.*
- 21.3 *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

## **22.0 Tables, Diagrams, Flowcharts and Validation Data**

- 22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.
- 22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.
- 22.3 Table 3, Technical Completeness / Accuracy Checklist
- 22.4 Table 4, Data Reviewers Checklist
- 22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

**APPENDIX:**

**ADDENDUM FOR USEPA SOW ILM05.2**

1. The CCV concentration must be different from the ICV.
2. The same CCV shall be used throughout analysis for an SDG.
3. Calibration standards must be within 5% of the standard concentration.
4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is  $\pm 30\%$ .
5. Spike samples at 1 ug/L for water.

**Table 1**

<b>Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>					
<b>Mercury by EPA 245.1, 7470A, SOW 4.1 &amp; 5.2</b>	<b>AQUEOUS MDL/DL (ug/L)</b>	<b>AQUEOUS LOD (ug/L)</b>	<b>AQUEOUS ERL/LOQ (ug/L)</b>	<b>AQUEOUS CRQL ILMO 4.1 (ug/L)</b>	<b>AQUEOUS CRQL ILMO 5.2 (ug/L)</b>
<b>Mercury</b>	0.080	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"> <li>Daily ICAL prior to sample analysis</li> <li>Low standard at the RL/LOD level</li> </ul>	<ul style="list-style-type: none"> <li>If more than one calibration standard is used, <math>r \geq 0.995</math></li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> </ul> <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"> <li>Re-run ICV</li> <li>Repeat ICAL</li> </ul>
Continuing calibration verification (CCV)	<ul style="list-style-type: none"> <li>After every 10 field samples and at the end of analysis sequence.</li> </ul>	<ul style="list-style-type: none"> <li><math>\pm 20\%</math> of true value</li> </ul>	<ul style="list-style-type: none"> <li>Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.</li> </ul>
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"> <li>Re-analysis to confirm the positive value</li> <li>Notify the PM for further action</li> <li>Re-prep of samples associated with the BLK</li> <li>NCR will be required for data reported</li> </ul>
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"> <li>Re-analyze to confirm failed.</li> <li>Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.</li> <li>NCR will be required for data reported</li> </ul>
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>

**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"> <li>• Initially per analyst prior to reporting data</li> <li>• Annually</li> <li>• Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>• Average percent recovery should be between 80-120%, with a 20% standard deviation.</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> </ul>
MDL Study	Once per year	<ul style="list-style-type: none"> <li>• Calculated value must be less than the Spike level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
LOD Verification	Every quarter	<ul style="list-style-type: none"> <li>• Parameter must be detected</li> <li>• the response must be 3-times the noise level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
LOQ Verification	Every quarter	<ul style="list-style-type: none"> <li>• Bias Requirement: Inorganics 50-150%</li> <li>• The LOQ value must be greater than the LOD value</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>

**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**

<b>Sample Number(s):</b>	
<b>Batch Number(s):</b>	<b>Sequence ID:</b>
<b>Method: 7470A/245.1 ( Mercury )</b>	

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did 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: \_\_\_\_\_

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**METALS: SOP 104      REVISION #: 19      EFFECTIVE DATE: 041110**

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**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: \_\_\_/\_\_\_/\_\_\_

Data Quality Manager: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Section Supervisor: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

## **Changes Summary**

### **Revision 19, 04/11/10**

- The SOP is an update from Revision 18 dated 03/25/10.

### **Revision 18, 03/08/10**

- The SOP is an update from Revision 17 dated 01/29/09.
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.
- Numerous improvements/modifications were made to this SOP. Details/specifications were added that require evaluation from start to finish.

## Table of Contents

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 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  $95^{\circ}\text{C}$  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 HNO<sub>3</sub>. Both HNO<sub>3</sub> and HCl must be of the reagent grade suitable for mercury determinations.  
**NOTE:** This reagent is required for use when USACE project samples are being digested.
- 10.1.3 Concentrated HCl.
- 10.1.4 Concentrated HNO<sub>3</sub>.
- 10.1.5 Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H<sub>2</sub>O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.

- 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 H<sub>2</sub>O; 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 HNO<sub>3</sub> (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 SnCl<sub>2</sub>. (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  $\text{KMnO}_4$  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  $\text{Cl}$  gas. The samples are now ready for analysis.
- 14.2.2.10 NOTE: Stannous Chloride (10.1.5) and 3%  $\text{HCl}$  (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 ( $\text{mg/kg}$  wet or  $\text{mg/kg}$  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**

<b>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 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>					
<b>Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 &amp; 5.2</b>	<b>SOLID/SOIL MDL/DL (mg/Kg)</b>	<b>SOLID/SOIL LOD (mg/Kg)</b>	<b>SOLID/SOIL ERL/LOQ (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)</b>	<b>SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)</b>
<b>Mercury</b>	0.013	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"> <li>Daily ICAL prior to sample analysis</li> <li>Low standard at the RL/LOD level</li> </ul>	<ul style="list-style-type: none"> <li>If more than one calibration standard is used, <math>r \geq 0.995</math></li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> </ul> <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"> <li>Re-run ICV</li> <li>Repeat ICAL</li> </ul>
CCV	<ul style="list-style-type: none"> <li>After every 10 field samples and at the end of analysis sequence.</li> </ul>	<ul style="list-style-type: none"> <li><math>\pm 20\%</math> for SW846-7471A&amp;B, <math>\pm 10\%</math> for 245.5 of true value</li> </ul>	<ul style="list-style-type: none"> <li>Follow guidelines for SOP QS05</li> </ul>
Closing CCV	<ul style="list-style-type: none"> <li>At the end of every sequence</li> </ul>	<ul style="list-style-type: none"> <li><math>\pm 20\%</math> for SW846-7471A&amp;B, <math>\pm 10\%</math> for 245.5 of true value</li> </ul>	<ul style="list-style-type: none"> <li>Follow guidelines for SOP QS05</li> </ul>
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"> <li>Re-analysis to confirm the positive value</li> <li>Notify the PM for further action</li> <li>Re-prep of samples associated with the BLK</li> <li>NCR will be required for data reported</li> </ul>
BS	One per prep batch	Most stringent criteria listed within the LIMS.	<ul style="list-style-type: none"> <li>Re-analyze to confirm failed.</li> <li>Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.</li> <li>NCR will be required for data reported</li> <li>Follow guidelines from SOP QS05</li> </ul>
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"> <li>Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.</li> </ul>
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>

**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"> <li>• Initially per analyst prior to reporting data</li> <li>• Annually</li> <li>• Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>• Average percent recovery should be between 80-120%, with a 20% standard deviation.</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> </ul>
MDL Study	Once per year	<ul style="list-style-type: none"> <li>• Calculated value must be less than the Spike level</li> <li>•</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
LOD Verification	Every quarter	<ul style="list-style-type: none"> <li>• Parameter must be detected</li> <li>• the response must be 3-times the noise level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
LOQ Verification	Every quarter	<ul style="list-style-type: none"> <li>• Bias Requirement: Inorganics 50-150%</li> <li>• The LOQ value must be greater than the LOD value</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>

**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**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method:</b> 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.

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**METALS: SOP 105      REVISION #: 16      EFFECTIVE DATE: 041110**

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**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 19<sup>th</sup> Edition 2340B;  
1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2**

**APPROVALS:**

Lab Director: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Data Quality Manager: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Section Supervisor: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

## **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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21. References
22. Tables, Diagrams, Flowcharts and Validation Data

## 1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19<sup>th</sup> 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 $\mu$ L; 5000 $\mu$ 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), HNO<sub>3</sub>. A method blank is digested and analyzed before a new lot number of HNO<sub>3</sub> 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% HNO<sub>3</sub> 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% HNO<sub>3</sub> 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 HNO<sub>3</sub> are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the 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 HNO<sub>3</sub> 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 19<sup>th</sup> 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 HNO<sub>3</sub>, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

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% HNO<sub>3</sub> / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ( $r \geq 0.998$ ). If a three point calibration curve is not required for the client samples being analyzed 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% HNO<sub>3</sub> / 5% HCl for at least 1 minute before the analysis of each sample.

14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7,  $\pm$  10% of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**

14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within  $\pm 10\%$  for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD)  $< 5\%$  from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be  $< 2x \pm MDL$ , for CLP  $< RL$ , for **DOD no analytes detected  $> \pm LOD$** . If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank  $< 1/10$  the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## 15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.1. Total hardness is reported from  $HNO_3$  preserved sample. The final concentration is calculated from the calcium and magnesium results as follows:  $Ca \text{ mg/L} \times 2.5 + Mg \text{ mg/L} \times 4.1 = \text{total Hardness in mg/L as } CaCO_3$ .

15.2. The instrument will generate data results in mg/L or  $\mu\text{g/L}$  (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ (ug/g or mg/kg)} = IR * DF * FED / SM$$

SR	=	Sample result
IR	=	Instrument result ( $\mu\text{g/L}$ )
DF	=	Dilution factor (post digestion)
FED	=	Final volume of digestate (L)
SM	=	Sample mass digested (g)

## 16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the supervisor for further processing and approval.

DOC LCS Preparation: See BS preparation under 10.7.1 through 10.7.3 above.

DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within  $\pm 50\%$ .

## 17. Pollution Prevention:

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

## 19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

## CORRECTIVE ACTIONS

### 19.1. INSTRUMENT RELATED

- 19.1.1. ICV not within  $\pm 10\%$  or  $\pm 5\%$  for 200.7
  - a. Is the problem with the solution?
    - i. Re-prepare or obtain new stock.

- b. Is the problem with the calibration?
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.
- 19.1.2. ICB not  $\pm$ MDL or within  $\pm$  3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**
- a. Is the problem with the solution?
    - i. Re-prepare
  - b. Is the problem with the calibration?
    - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.
- 19.1.3. Check standards not within  $\pm$  5%
- a. Is the problem with the solution?
    - i. Re-pour, re-prepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.4. CLP only-CRI not within  $\pm$  20% (Internal QC, only required for CLP work).
- a. Is the problem with the solution?
    - i. Re-prepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes  $<\pm$ LOD.**
- a. Is the problem with the solution?
    - i. Re-prepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.6. IFB not within  $\pm$  20%
- a. Is the problem with the solution?
    - i. Re-prepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
- 19.1.7. CCV not within  $\pm$  10%
- a. Is the problem with the solution?
    - i. Re-prepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

- 19.1.8.. CCB not  $\pm 2 \times \text{MDL}$  or CRDL for CLP, DOD no analytes detected  $> \pm \text{LOD}$ .
  - 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  $\pm \frac{1}{2} \text{RL}$  and  $\pm \text{RL}$  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  $> 5 \times \text{CRDL}$ ) or  $\pm$  the CRDL (if either are  $< 5 \times \text{CRDL}$ ).
  - 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

<b>Table 1 Water</b>				
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
<b>Table 1 TCLP</b>				
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

<b>Table 1 Soil</b>				
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**

<b>METAL</b>	<b>WAVELENGTH</b>
<b>Aluminum</b>	<b>396.1</b>
<b>Antimony</b>	<b>206.8</b>
<b>Arsenic</b>	<b>189.0</b>
<b>Barium</b>	<b>233.5</b>
<b>Beryllium</b>	<b>313.0</b>
<b>Boron</b>	<b>249.7</b>
<b>Cadmium</b>	<b>228.8</b>
<b>Calcium</b>	<b>317.9</b>
<b>Chromium</b>	<b>267.7</b>
<b>Cobalt</b>	<b>228.6</b>
<b>Copper</b>	<b>324.7</b>
<b>Iron</b>	<b>261.1</b>
<b>Lead</b>	<b>220.3</b>
<b>Magnesium</b>	<b>279.0</b>
<b>Manganese</b>	<b>257.6</b>
<b>Molybdenum</b>	<b>202.0</b>
<b>Nickel</b>	<b>231.6</b>
<b>Potassium</b>	<b>766.4</b>
<b>Selenium</b>	<b>196.0</b>
<b>Silver</b>	<b>328.0</b>
<b>Sodium</b>	<b>589.5</b>
<b>Strontium</b>	<b>421.5</b>
<b>Thallium</b>	<b>190.8</b>
<b>Tin</b>	<b>189.9</b>
<b>Titanium</b>	<b>334.9</b>
<b>Vanadium</b>	<b>292.4</b>
<b>Zinc</b>	<b>206.2</b>

**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"> <li>once per calibration</li> </ul>	<ul style="list-style-type: none"> <li>IFA less than LOD if not verified contamination of standard. IFB must be within <math>\pm 20\%</math>.</li> </ul>	<ul style="list-style-type: none"> <li>Check IEC corrections for metals in the IFA.</li> </ul>
Calibration Curve	<ul style="list-style-type: none"> <li>Prior to analyzing any samples</li> <li>A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard.</li> <li>Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C).</li> </ul>	<ul style="list-style-type: none"> <li>Linear calibration Corr. of 0.998</li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-evaluate curve mix and makeup</li> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> <li>Re-prepare the curve standards</li> </ul> <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"> <li>Must be in the range 90 to 110% for 6010B&amp;C, or 95 to 115% for 200.7.</li> </ul>	<ul style="list-style-type: none"> <li>Re-analyze an ICV from a different source</li> <li>Re-prepare and re-analyze the ICV</li> <li>Re-calibrate and verify standard preps and sources</li> </ul>
CCV	<ul style="list-style-type: none"> <li>At the beginning of every sequence</li> <li>For every 10-client samples</li> </ul>	<ul style="list-style-type: none"> <li>Must be in the range 90 to 110%</li> </ul>	<ul style="list-style-type: none"> <li>Samples must be reanalyzed if possible, if not samples are flagged with a "Q".</li> </ul>
Closing CCV	<ul style="list-style-type: none"> <li>At the end of every sequence</li> </ul>	<ul style="list-style-type: none"> <li>Must be in the range 90 to 110%</li> </ul>	<ul style="list-style-type: none"> <li>Samples must be reanalyzed if possible, if not samples are flagged with a "Q".</li> </ul>
BLK	One per prep batch	<ul style="list-style-type: none"> <li>Must be less than <math>\frac{1}{2} \pm \text{RL}</math>.</li> </ul>	<ul style="list-style-type: none"> <li>Re-analysis to confirm the positive value</li> <li>Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers</li> <li>If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action</li> <li>Re-prepare of samples associated with the MB</li> <li>NCR will be required for data reported</li> <li>Final Report data flagging will be required</li> </ul>

**Table 2 - Method Quality Control Requirements Summary**

<b>QC Check</b>	<b>Minimum Frequency / Requirements</b>	<b>Acceptance Criteria</b>	<b>Corrective Action for Failures / Data Useability</b>
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"> <li>• Rerun to confirm problem.</li> <li>• All samples associated with the LCS must be re-digested, reanalyzed if possible.</li> <li>• NCR will be required for data reported</li> <li>• If samples cannot be re-digested or re-analyzed Final Report data flagging will be required</li> </ul>
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"> <li>• Initially per analyst prior to reporting data</li> <li>• Annually</li> <li>• Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>• Must meet the criteria of the BS for average accuracy</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or</li> <li>• Re-analysis</li> </ul>
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

<b>ANALYST DATA REVIEW CHECKLIST Sample Number(s):</b>				
<b>Batch Number(s):</b>				
<b>Method: 6010B or 6010C ( ICP )</b>				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did 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:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**ORGANICS: SOP 145 REVISION #: 07 EFFECTIVE DATE: 20100325**

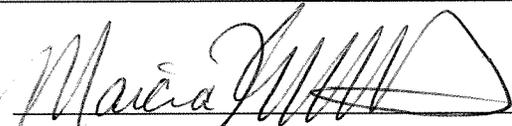
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**DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING  
THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX  
AS18 COLUMN**

**References:  
USEPA METHOD 300.0/ SW846 Method 9056**

**APPROVALS:**

Lab Director:  Date: 3/30/10

Data Quality Manager:  Date: 3/30/10

Section Supervisor:  Date: 3/30/10

**Changes Summary:**

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness.
- All references to analysis of ortho-phosphorus by this method have been removed.

**DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING  
THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX  
AS18 COLUMN**

**References:  
USEPA METHOD 300.0/ SW846 Method 9056**

**I. SCOPE AND APPLICATION:**

1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes.

**PART A.--Common Anions**

Chloride	Nitrate	Fluoride	Sulfate
Nitrite	Bromide		

2. Single laboratory Method Detection Limit for the above analytes is listed in Tables 1A, 1B and 1C from method 300.0. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.
  - A. In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more than a 5 nS noise/drift per minute of monitored response over the background conductivity.
3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.
4. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.
5. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

**II. SUMMARY OF METHOD**

1. A small volume of sample, 50 uL for Part A is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

**III. DEFINITIONS**

1. **ANALYSIS BATCH** -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices (MS/MSD)). The analysis batch must include an Initial Calibration Check Standard (CCV), and End Calibration Check Standard (ending CCV), Laboratory Reagent Blank (BLK), and a

Laboratory Fortified Blank (BS). Within an ANALYSIS BATCH, for every group of **ten** field samples at least one Laboratory Fortified Matrix (MS) and either a Field Duplicate or a Laboratory Duplicate must be analyzed after the tenth field sample analysis. MSD does not count as a laboratory duplicate for anions.

2. **CALIBRATION STANDARD (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
  - A. **INITIAL CALIBRATION STANDARDS** -- A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions.
  - B. **INITIAL CALIBRATION CHECK STANDARD** -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.
  - C. **CONTINUING CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after every tenth field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.
  - D. **END CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.
3. **FIELD DUPLICATES (FD)** --Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
4. **INSTRUMENT PERFORMANCE CHECK SOLUTION (ICV)** -- A solution of one or more method analytes, surrogates or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
5. **LABORATORY DUPLICATE (DUP)** -- Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of sample and DUP1 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.
6. **LABORATORY FORTIFIED BLANK (BS)** --An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
7. **LABORATORY FORTIFIED SAMPLE MATRIX (MS)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the

laboratory. The MS and or MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

8. **LABORATORY REAGENT BLANK (BLK)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
9. **LINEAR CALIBRATION RANGE (LCR)** -- The concentration range over which the instrument response is linear.
10. **MATERIAL SAFETY DATA SHEET (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
11. **METHOD DETECTION LIMIT (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
12. **MINIMUM REPORTING LEVEL (MRL)** -- The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.
13. **PERFORMANCE EVALUATION SAMPLE (PE)** -- A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.
14. **QUALITY CONTROL SAMPLE (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
15. **STOCK STANDARD SOLUTION (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

#### **IV. INTERFERENCES**

1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window

of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes' retention times.

- A. A direct chromatographic coelution may be solved by changing columns, eluant strength, modifying the eluant with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.
- B. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluant.
- C. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.
  - 1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response ratios between the target analytes is observed.
- D. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.
- E. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.
- F. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.
- G. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate (retention time of ~9.0 min.) indicates the end of a

chromatographic run. A run time of 12 minutes is recommended to allow for the proper elution of any potentially interferrant late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

## 2. **SAFETY**

- A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

## V. **EQUIPMENT AND SUPPLIES**

1. Ion Chromatograph (IC) – Analytical system complete with eluant generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500 or equivalent. (See letter from EPA to Dionex on discussion of alternate hydroxide eluant for anions. Also since hydroxide eluant cannot be run on the traditional column Dionex AS18, 4mm (P/N 060549) or equivalent should be used.
  - a. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.
  - b. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met.
    - i. When a 4 mm column is employed, the injection volume should be 50 uL.
    - ii. Comparable results can be attained using the Dionex, AS17, 4 mm column.
2. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 53946). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.
  - a. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical

Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.

3. Detector--Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in the Quality Control section of this SOP.
4. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2 or equivalent is used by Empirical Laboratories.
5. Analytical balance--Mettler Used to accurately weigh target analyte salt for stock standard preparation ( $\pm 0.1$  mg sensitivity).
6. Micro beakers -- Plastic, disposable - used during sample preparation.
7. Syringes--Plastic, disposable, 10 mL - used during sample preparation.
8. Eppendorfs with variable settings- 1mL and 5 mL. Must be calibrated quarterly.
9. Bottles -- High density polyethylene ( HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions.
10. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Gelman IC Acrodisc, PN 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.

NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.

11. Autosampler PolyVials 5-mL size, with filtercaps, 250 each --Dionex cat log # 38141.
12. Shaker for use when extracting soil samples.
13. Centrifuge to aid in separation after extraction.
14. Centrifuge tubes--50 mL capacity

## VI. REAGENTS AND STANDARDS

1. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.
2. A system or apparatus which automatically generates the hydroxide eluant (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluant.
3. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors.

**NOTE:** Stability of standards: Stock standards for most anions are stable for at least 6 months when stored at 4 °C. Dilute working standards should be prepared monthly.

## VII. SAMPLE COLLECTION, PRESERVATION AND STORAGE

1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.
2. Sample preservation and holding times for the anions that can be determined by this method are as follows:

### PART A: Common Anions

<u>Analyte</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
<b>Nitrate-N</b>	<b>Cool to 4 °C</b>	<b>48 hours</b>
<b>Nitrite-N</b>	<b>Cool to 4 °C</b>	<b>48 hours</b>
Sulfate	Cool to 4 °C	28 days

3. When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the addition of the EDA preservative at time of sample collection.

## VIII. QUALITY CONTROL

1. The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.
2. INITIAL DEMONSTRATION OF PERFORMANCE
  - A. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.
  - B. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within  $\pm 10\%$  of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

- C. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [ t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

- D. MDLs should be determined every 6 months or at least annually, when a new operator begins work or whenever there is a significant change in the background, or instrument response. MDL check samples are used in connection with confirming that the MDL determined is legitimate and to monitor the instrument sensitivity periodically. MDL checks are analyzed whenever a new MDL is generated and at a minimum quarterly to monitor for any shifts in sensitivity.

### 3. ASSESSING LABORATORY PERFORMANCE

- A. Laboratory Reagent Blank (BLK) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL (**For DOD QSM Ver. 3 no analytes detected  $\geq \frac{1}{2}$  RL or for common lab contaminants no analyte detected  $\geq$  RL**) indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.
- B. Laboratory Fortified Blank (BS)—The BS should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the MS/MSD. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.
- i. Control Limits for the BS are 90 to 110%.
  - ii. The laboratory uses the BS to assess laboratory performance against the required control limits listed in the QC section. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\begin{aligned}\text{UPPER CONTROL LIMIT} &= x + 3S \\ \text{LOWER CONTROL LIMIT} &= x - 3S\end{aligned}$$

The optional control limits must be equal to or better than those listed in the QC section ( $\pm 10\%$ ). After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be on file and be available for review.

- i. Instrument Performance Check Solution (ICV) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard is 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluant (or when the KOH cartridge is changed) is prepared but if shifts of more that 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluant, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the ICV to provide evidence of an analytical column's vitality.

#### 4. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

- A. Laboratory Fortified Sample Matrix (MS) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The MS sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify the MS be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.
  - i. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.
  - ii. The MS should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the MS must be fortified no greater than five times the lowest calibration level which as outlined in this method is the minimum reported level (MRL). For example, if chloride is not detected in

any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest MS fortified concentration allowed is 25.0 ug/L.

- iii. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{S} \times 100$$

where, R = percent recovery.  
 C<sub>s</sub> = fortified sample concentration  
 C = sample background concentration  
 S = concentration equivalent of analyte added to sample.

- iv. Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 80 to 120%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 80 –120%.
- v. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a “matrix induced bias” qualifier.

B. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

- i. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I<sub>c</sub>) and duplicate quantitated concentration (D<sub>c</sub>) using the following formula,

$$RPD = \frac{(I_c - D_c)}{[(I_c + D_c)/2]} \times 100$$

- ii. Duplicate analysis acceptance criteria

<u>Concentration range</u>	<u>RPD Limits</u>
MRL to 10xMRL	± 20%
10xMRL to highest calibration level	± 10%

- iii. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.
- C. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.
- D. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluants, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.
- E. The laboratory adopts additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participate in relevant performance evaluation sample studies.

## 5. CALIBRATION AND STANDARDIZATION

- A. Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1C for a 4-mm column.
  - i. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.
  - ii. For an individual calibration curve, a minimum of eight calibration standards is required for a curve.
- B. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are calibrated from 0.5-200 mg/L; fluoride, nitrate, and nitrite from 0.05-20 mg/L.
- C. Using a 4mm column, inject 50 uL (Part A) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD)), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.



vii. Once changes have been made go to main menu, batch, processing, input tab and choose schedule, then output tab and check update raw data files. (**DO NOT CHECK UPDATE CALIBRATION STANDARDS** or it will erase all the changes that were just made). Then click OK then F5.

viii. Go to main menu – optimize, open the QC files that were ran after curve and check. ICV must be  $\pm 10\%$ .

ix. When curve is complete, gather raw data before and after and print out the curve. To do this open a blank EXCEL sheet, then go to main menu – method – open current method, go to each analyte and press ALT PRINT SCREEN then paste into the EXCEL file. A copy of all this will go to the reporting department.

## 6. **PROCEDURE**

A. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.

B. Check system calibration daily and, if required, recalibrate as necessarily.

### C. Sample Preparation

i. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.

D. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45  $\mu\text{m}$  particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.

E. Using a 4 mm column, inject 50  $\mu\text{L}$  of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.

F. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

G. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to  $\pm 25\%$  the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required.

H. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be

affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.

- I. Should more complete resolution be needed between any two coeluting peaks, the eluant can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.
  - i. Eluant dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely affect the MDLs for each analyte.

## 7. DATA ANALYSIS AND CALCULATIONS

- A. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.
- B. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).
- C. Report results for Part A anions in mg/L.
- D. Report  $\text{NO}_3^-$  as N

### Traceability

A record shall be maintained on all reference materials within Element. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in Element as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, date opened, and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded within Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

## IX. INSTRUMENT INFORMATION

Analyst should confirm the following:

### 1. Start Up routine for instrument is as follows:

- a. Turn Power on to autosampler, conductivity detector, eluant generator and pump in any order.
  - b. Turn on the Helium gas supply~ 80 to 100. Ensure gas lines to bottles not in use are off. Ensure air supply to the injection valve is on.
  - c. Close the vent valves on sparging bottles and allow head pressure to build for a few moments. DI H2O bottles should be 7 to 10 psi.
  - d. Open the eluant supply valve(s) for the bottles in use. Check for sputter after eluant flow has started.
  - e. Inject 5 mL of DI water into the pump head and clean the pump head. Also check the frits on the guard column and change if necessary. If necessary to change guard column frits check primary column frits as well.
  - f. Load the auto sampler cartridges and put autosampler into RUN state.
  - g. To vent airlocks, Run eluant with pump on and SRS off, open waste valve-> bottom door top black knob, just for a few seconds and close it.
  - h. Turn on the SRS power. NOTE: NEVER TURN ON THE SRS POWER SUPPLY WITHOUT THE PUMP GOING FIRST!!! Use either LOCAL/DIRECT CONTROL to enter commands at keypads, or REMOTE/DIRECT CONTROL to use the direct control option from the RUN menu within PEAKNET.
  - i. After System has come to equilibrium, load sequence and run.
2. Shut Down routine for instrument is as follows:
- a. If the instrument is not going to be operated for a period of time, run deionized water through the eluant lines for ~ 30 minutes to an hour to rinse the lines.
  - b. Stop the OFF/ON pump and then select SRS-OFF.
  - c. Close gas supply valves and eluant valves on the eluant bottles. Turn off the supply. Is not necessary to vent the eluant bottles.
  - d. Power down the modules in any order.
3. General Sample Loading and Run Set-up.
- a. Enter Peak-net Software from Desktop.
  - b. Loading a Run: Click on Schedule. The headings within the Schedule Editor are SAMPLE, SAMPLE TYPE, LEVEL, METHOD and DATA FILE.
    - i. Name each sample under the SAMPLE heading column. (, CCV, CCB, CRL, BS, BLK, sample #'s, etc.)
    - ii. SAMPLE TYPE is sample unless loading a calibration curve.

- iii. LEVEL designations are used only when assigned to a calibration curve.
- iv. Enter method name under METHOD heading. In most cases, date of most recent calibration in Anions Method file will be used.
- v. Enter the date under the DATA FILE heading. The program will then sequentially assign the data file names based on the date.
- vi. All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.
- vii. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. It is not necessary to include a vial in the corresponding position in the autosampler. Sample type is Sample, and Method is entered as <pumpoff.met>.

**Typical run-log:**

1 Blank  
 2 CCV  
 3 CCB  
 4 CRL1  
 5 CRL2  
 6 BS1  
 7 BLK1  
 8 Sample  
 9 Sample @ 10X  
 10 Sample @ 50X  
 11 Sample  
 12 Sample  
 13 Sample  
 14 CCV  
 15 CCB  
 16 Sample  
 17 Sample  
 18 Sample  
 19 Sample  
 20 Sample  
 21 Sample  
 22 Batch #-MS1  
 23 Batch #-MSD1  
 24 Batch #- DUP1  
 25 Sample @ 10X  
 26 Sample  
 27 Sample  
 28 Sample  
 29 CCV  
 30 CCB  
 31 Sample  
 32 Sample

33	Sample
34	Sample
35	Sample
36	Batch #-MS2
37	Batch #-MSD2
38	Batch #- DUP2
39	Sample
40	Sample
41	Sample
42	Sample
43	CCV
44	CCB
45	Sample
46	Sample
47	Sample
48	CCV
49	CCB
50	pumpoff

- c. Save a schedule under File/Save as, using the date as the title of the Schedule.
- d. When saved, exit out of Schedule Editor.
- e. Load autosample cartridges in the same order as the scheduled run. After putting the cartridges in the autosampler, switch the autosampler to RUN using the Hold/Run button.
- f. Assuming that the Pump is equilibrated with steady eluant baseline/uniform conductivity, enter into the RUN page.
- g. Go to File to Open Method. Open correct method <date> of most recent calibration. This will begin pumping eluant at 1.0 mL/minute and turn on the SRS pump at 300  $\mu$ amps voltage.
- h. Next, go to file to Open Schedule. Open newly created schedule for the day.
- i. When Method and Schedule are opened, go to Run and click on Start. The autosampler will inject into the first sample and the run should continue until completion.

## **X. POLLUTION PREVENTION**

- A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

- B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- C. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

## **XI. WASTE MANAGEMENT**

- A. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

## **XII. CORRECTIVE ACTIONS**

### **A. INSTRUMENT RELATED**

- 1. ICV not within  $\pm 10\%$ 
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration. Recalibrate thru analysis of appropriate standards and recheck ICV.
- 2. CCV not within  $\pm 10\%$ 
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten samples according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalysed.

3. CCB not  $> \pm$  MDL (USACE) (**For DOD QSM Ver. 3 no analyte detected  $> 2x$ MDL, frequency- beginning and ending a run and every 10 samples**) or  $\pm$ RL or CRDL for others and CLP
  - a. If the CCB is biased high.
    - i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.
    - ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.
  - b. If the CCB is biased low.
    - i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
    - ii. All other samples must be reanalyzed after the problem is corrected.
4. BS not within our in-house generated control limits (or  $\pm 10\%$ ).
  - a. If the problem is with the instrument.
    - i. Reanalyze when instrument is in control.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-extracted or the data will be qualified on the final report.

### C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 20\%$ 
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

### XIII. SOURCES/REFERENCES:

1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18<sup>th</sup> Edition of Standard Methods (1992).
2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.
3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, December, 1981.

4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).
5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.
6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.
7. USEPA Methods 300.0; *Method for Determination of Inorganic Substances*(EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).
8. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B.

## ANALYST DATA REVIEW CHECKLIST

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: EPA 300.0 Anions by Ion Chromatography</b>
Instrument is a Dionex DX-500 system. Equipped with Guard Column, Analytical Column, Conductivity Suppressor, Conductivity Detector, and Eluant Generator.

<u>QA/QC Item</u>	<u>Yes</u>	<u>No</u>	<u>NA</u>	<u>Second Level Review</u>
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did CRL meet control limits?	_____	_____	_____	_____
6. Did BS, Laboratory Fortified Blank or blank spike meet control limits?	_____	_____	_____	_____
7. Did MS/MSD meet control limits? Did Duplicate meet control limits?	_____	_____	_____	_____
8. Was the Blank below the project required detection limits?	_____	_____	_____	_____
9. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
10. Were samples analyzed for Nitrate (as N) and Nitrite (as N) done within the 48-hr holding time?	_____	_____	_____	_____
11. Sample preparation information is correct and complete.	_____	_____	_____	_____
12. Were all samples filtered through a 0.45µm filter?	_____	_____	_____	_____
13. Analytical results are correct and complete.	_____	_____	_____	_____
14. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
15. Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
16. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
17. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

**ADDITIONAL INFORMATION:**

**All manual integrations of standards have been checked and confirmed by supervisor or qualified personnel.**

\_\_\_\_ yes \_\_\_\_ no      **Data confirmed by** \_\_\_\_\_ **Date:** \_\_\_\_\_

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**SULFIDE  
METHOD 376.1 and STANDARD  
METHODS SM4500S F(19<sup>th</sup> ED)  
(TITRIMETRIC, IODINE)  
WITH SAMPLE PRETREATMENT TO  
REMOVE INTERFERING  
SUBSTANCES OR TO CONCENTRATE  
THE SULFIDE**

---

**SOP NUMBER:** SOP-153

**REVISION NUMBER:** 3

**APPROVED BY:**

*Betty DeVillo*  
SECTION MANAGER

*Randy Ward*  
TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 06/24/08

**DATE OF LAST REVIEW:** 05/27/09

**Empirical Laboratories, LLC**

**SULFIDE**  
**METHOD 376.1 and STANDARD METHODS SM4500S F(19<sup>th</sup> ED)**  
**(TITRIMETRIC, IODINE)**  
**WITH SAMPLE PRETREATMENT TO REMOVE INTERFERING**  
**SUBSTANCES OR TO CONCENTRATE THE SULFIDE**

**I. SCOPE OF APPLICATION:**

- A. This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.
- B. Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class.)
- C. This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.

**II. SUMMARY OF METHOD:**

Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate.

**III. SAMPLE HANDLING AND PRESERVATION**

- A. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.
- B. Samples are taken in glass bottles with stopper; preferably 500 to 1000 mL. Usually we use 300 mL (BOD bottle). Preserve with zinc acetate 2N and 6N NaOH. There should be no air-space in the container.
- C. The holding time for sulfides preserved in this manner is 7 days.

**IV. INTERFERENCES:**

The iodometric method suffers interferences from reducing substances that react with iodine, including thiosulfate, sulfite and various organic compounds, both solid and dissolved. Interferences due to sulfite, thiosulfate, iodide and many

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other soluble substances are eliminated by first precipitating ZnS in the samples, removing the supernatant, and replacing it with distilled water.

**V. EQUIPMENT/APPARATUS:**

- A. 10 mL burette
- B. Glass, stoppered bottle of 500 to 1000 mL (300 mL BOD bottles)
- C. Vacuum pump
- D. Magnetic stirrer with Teflon coated stirring bars
- E. Buchner funnel
- F. 500 mL Erlenmeyer flask

**VI. REAGENTS:**

- A. Zinc acetate, 2N: dissolve 220 grams Zn (C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> · 2 H<sub>2</sub>O in 870 mL D.I. water; this makes 1 liter solution.
- B. Sodium Hydroxide, 6N: dissolve 240 grams of NaOH in about 600 mL of D.I. water. Dilute to 1 liter.
- C. Hydrochloric acid, 6N: add 250 mL concentrated HCl to 250 mL D.I. water, mix well.
- D. Standard iodine solution, 0.025N: dissolve 20 to 25 grams KI in a little water and add 3.2 grams iodine. After iodine has dissolved, dilute to 1000 mL in a volumetric flask. Standardize against 0.0250N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, using thyodene as indicator. **(Purchased commercially)**
- E. Standard sodium thiosulfate titrant solution, 0.025N: dissolve 6.205 grams Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O in distilled water. Add 1.5 mL 6N NaOH or 0.4 grams solid NaOH and dilute to 1000 mL. Standardize with bi-iodate solution. (May be purchased commercially.)
- F. Standard potassium bi-iodate solution, 0.0250N: dissolve 812.4 mg KH(IO<sub>3</sub>)<sub>2</sub> in distilled water and dilute to 1000 mL. Standardization of thiosulfate--dissolve approximately 2 grams KI, free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6N H<sub>2</sub>SO<sub>4</sub> or a few drops of concentrated H<sub>2</sub>SO<sub>4</sub> and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding 1 scoop thyodene toward end of titration, when a pale straw color is reached. When the solutions are of equal strength, 20.00 mL 0.0250N Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> should

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be required. If not, adjust the  $\text{Na}_2\text{S}_2\text{O}_3$  solution to 0.0250N using  $N_1V_1 = N_2V_2$  equation.

G. Thyodene.

**VII. PROCEDURE**

A. Pretreatment:

1. If sample has not been preserved with zinc acetate and NaOH, place 3 to 5 pasteur pipettes of 2N zinc acetate solution into a 500 mL glass bottle, fill with sample, and add 10 drops 6N NaOH solution. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously about a transverse axis. Vary volume of reagents added according to sample so that the resulting precipitate is not excessively bulky and settles readily. Add enough NaOH to produce a pH above 9.
2. Let precipitate settle for 30 minutes. The treated sample is relatively stable and can be held for several hours. However, if much iron is present, oxidation may be fairly rapid. Holding time for this sample is 7 days.

B. Preparation and Titration of Sample:

1. Mark meniscus of sample volume on side of bottle so you can measure sample volume. Filter precipitate through glass fiber filter paper 11.0 to 12.5 cm in a Buchner funnel. Save the filter and all precipitate and discard filtered sample.
2. Measure exactly, amount of standard iodine solution (estimated to be an excess over the amount of sulfide present in the sample--usually 2 to 10 mL) into a 500 mL Erlenmeyer flask. Add distilled water, if necessary, to bring volume and iodine solution to 20 mL.
3. Add 2 mL 6N HCl.
4. Place filter with precipitate, making sure you wipe sides of Buchner funnel to get any precipitate clinging to the sides, into bottle with iodine solution and acid. Add 200 mL D.I. water.
5. Fill a 50 mL Burette with 0.0250 N sodium thiosulfate solution.

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6. Put a small stirring bar in sample and place on magnetic stirrer. Stir bar turning slowly.
7. Titrate slowly with sodium thiosulfate titrant adding 1 scoop thyodene reagent toward the end of the titration, when a pale straw color is reached. The sample will go from straw yellow to a dark blue when thyodene is added. It should take only 2 or 3 drops of titrant to bring sample to the endpoint of clear at this point. Record mL of titrant used. Sample will turn back blue but the first change from blue to clear is the endpoint.
8. Do a blank with each set of samples taken D.I. water through the entire procedure the same as the samples including pretreatment. D.I. water is usually ~1.0 mg/L sulfide.
9. Discard titrated sample. Rinse out bottle and measure volume of sample used by filling bottle to the calibration mark on the side of bottle with water and pouring water into a graduate cylinder.

**VIII. CALCULATIONS:**

One milliliter 0.0250N iodine solution reacts with 0.4 mg S<sub>2</sub>-:

$$\text{Mg S}_2\text{-/L} = \frac{(AXB) - (CXD)}{\text{Sample}} \times 16000 \times (\text{Ratio of final to mL initial volume})$$

where:

- A = mL standard iodine solution
- B = normality of iodine solution
- C = mL Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution
- D = normality of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution
- Final volume = 200 mL
- Initial volume = measured volume of original sample

**IX. QUALITY CONTROL:**

- A. Analyze a duplicate and second source check with each analytical batch. The second source check standard can be purchased commercially.

**X. CORRECTIVE ACTIONS**

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- A. If the LCS fails (exceeds 80-120 %). Contact Lab supervisor.

**XI. HEALTH AND SAFETY**

- A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
- B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- B. MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

**XII. POLLUTION PREVENTION**

- A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.
- B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- C. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

**XIII. WASTE MANAGEMENT**

**Empirical Laboratories, LLC**

- A. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

**XIV. METHOD PERFORMANCE**

See Methods 376.1 and Standard Methods SM4500S F.

**TOTAL ALKALINITY , CARBONATE,  
BICARBONATE**

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**METHOD  
USEPA 310.1, SM2320B**

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**SOP NUMBER: SOP-154**

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**REVISION NUMBER: 5**

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**APPROVED BY:**

*Betty DeVill*  
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**TECHNICAL DIRECTOR**

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**EFFECTIVE DATE: 05/27/09**

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**DATE OF LAST  
REVIEW: 05/27/09**

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## **TOTAL ALKALINITY, CARBONATE, BICARBONATE Method EPA 310.1 & Standard Methods 2320B**

### **I. SCOPE AND APPLICATION**

- A. This method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes. Soils are leached 10 grams to 100 mLs and the analysis performed on the leachate.
- B. The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 10 mL.

### **II. SUMMARY OF METHOD**

An unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The calculation for total alkalinity (in calculation section of this SOP) is then used to calculate.

When the sample is being analyzed for phenolphthalein alkalinity, carbonate, bicarbonate and total alkalinity, method 2320B is used. With this method, an unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample is then titrated to a pH exactly 0.3 pH units lower and the calculation for 2320B (in calculation section of this SOP) is used to calculate the samples for phenolphthalein alkalinity, total alkalinity, carbonate and bicarbonate results. See note at the end of section VIII (Procedure) after step N for samples with pH greater than 8.3.

### **III. DEFINITIONS**

1. **Preparation Blank (PB)**- Laboratory reagent water that is treated exactly as a sample including exposure to all glassware, equipment (pH probe) and reagents that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.
2. **Laboratory Control Sample (LCS)**- An aliquot of reagent water or other blank matrices to which known quantities of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is given a

unique identifier so that it is traceable to its source and concentration and expiration date.

3. **Analysis Batch-** An analysis batch is a group of twenty field samples, a preparation blank, a laboratory control sample and a sample and/or laboratory control sample duplicate.
4. **Sample Duplicate-** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of sample one and sample two indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.
5. **Method Detection Limit (MDL)-** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
6. **Performance Evaluation Sample (PE)-** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

#### IV. SAMPLE HANDLING AND PRESERVATION

- A. No preservation necessary except to keep chilled to 4°C until sample is analyzed. Do not open sample bottle until analysis.
- B. The holding time for these samples is 14 days. Example: If sampled on November 1 at 10 a.m., analysis must be performed by November 14 at 10 a.m.

#### V. INTERFERENCES

- A. Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
- B. For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in *Annual Book of ASTM Standards*, Part 31, "Water," p. 115, D-1067, Method D (1976).

- C. Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.

## VI. EQUIPMENT/APPARATUS

- A. pH meter that uses a glass electrode and can be read to 0.01 pH units. The analyst will note on the data which pH meter (either the Corning 240 or Orion 420A) is used.
- B. 50 mL disposable beakers with wide enough mouths to allow room for burette tip and pH probe.
- C. 10 mL Class A microburette.

## VII. REAGENTS

- A. Sodium carbonate solution, approximately 0.05N: Place  $2.5 \pm 0.2$  g (to nearest mg)  $\text{Na}_2\text{CO}_3$  (dried at  $250^\circ\text{C}$  for 4 hours and cooled in desiccator) into a 1-liter, Class A, volumetric flask and dilute to the mark. The  $\text{Na}_2\text{CO}_3$  solution must be disposed of after one week.
- B. Standard Acid (sulfuric or hydrochloric), 0.1N (high titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and kept on file when a purchased solution is used.** Dilute 3.0 mL concentrated  $\text{H}_2\text{SO}_4$  or 8.3 mL concentrated HCl to 1 liter with distilled water. Dilute 40 mL of 0.05N  $\text{Na}_2\text{CO}_3$  solution to 100 mL with deionized water and titrate potentiometrically with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). This standardization must be done at least every three months. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where: A = g  $\text{Na}_2\text{CO}_3$  weighed into 1 liter  
B = mL  $\text{Na}_2\text{CO}_3$  solution  
C = mL acid used to inflection point

- C. Standard Acid (sulfuric or hydrochloric), 0.02N (low titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and**

**kept on file when a purchased solution is used.** Dilute 200.0 mL of 0.1000 N Standard Acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 mL 0.05N Na<sub>2</sub>CO<sub>3</sub> solution every three months as above.

## VIII. PROCEDURE

- A. Write down time test started.
- B. Fill 10 mL microburette with Standard Acid.
- C. Pick titrant according to estimated total alkalinity. For example, a drinking water or groundwater sample would probably use the 0.020 N titrant and a wastewater sample would probably use the 0.10 N titrant. Historical data is very useful for this.

A sample size of 25 mL is usually appropriate. If you use less than 1 mL of your high titrant, then you must titrate a new sample using low titrant. Using less than 1 mL of your low titrant is valid. When the samples are soils a 12 gram portion diluted to 120 mLs is used.

- D. Sample size should be such that a sufficiently large volume of titrant is used (1 to 10 mL titrant).
- E. Standardize and calibrate the pH meter according to laboratory procedures as explained. Using the Corning 240 pH meter, first calibrate the meter by putting the probe (which has been filled with the correct filling solution) in 7 buffer, setting the pH at 7; then in 4 buffer, setting the pH at 4, and then checking 7 again to make sure it still reads 7, if an adjustment is made to the 4 buffer then check that buffer again as well. The calibration buffers must be within  $\pm 0.05$  pH units of the true value. Then check the 10 buffer. The reading should be within  $\pm 0.10$  pH units. If not, recalibration is necessary. Record this information in the appropriate log book. If automatic temperature compensation is not provided, make titration at  $25 \pm 2^\circ\text{C}$ . Check the buffer every 3 hours after calibration. The reading should be within  $\pm 0.20$  pH units.
- F. Carefully pour 25 mLs into disposable beaker by gently pouring down the side of the vessel so as to have the least aeration to sample as possible. Place a small magnetic stirring bar in vessel and start magnetic stirrer at medium to slow stirring.

Note 1: When soils are being analyzed 12 grams is weighed into a 120 mL bottle and diluted to 120 mLs. Place in the shaker for one hour. Mix the

sample well and use 25 mLs to analyze. Make sure you get a representative sample for analysis.

Note2: Where sample volume is adequate when using low titrant, a sample volume of 100 to 200 mL should be used and titration should be performed using a 10 mL microburette.

- G. Place pH probe (which has been rinsed with DI water and patted dry with a Kimwipe) in the sample such that the probe tip is not touching the sides or bottom of the flask or beaker. If the probe has a protective cover, this is not a consideration.
- H. Make sure there are no air bubbles at the bottom of filled burette. Wipe tip of burette so that no extra drops are clinging to it. Place tip of burette into mouth of vessel so that it is above the surface of the sample but is not touching the sides of the flask and drops can go nowhere but into the sample (e.g., drops from burette are not going onto pH probe or walls of flask but directly into sample).
- I. Titrate a blank and an LCS first. This will let you know that the normalities of titrant are correct. If the result is out of the acceptable range of the LCS, run a duplicate LCS. If still out of range, find another second source and if still incorrect, restandardize titrant. First double-check titrant normality.
- J. Record sample pH after reading is stable for 5 to 10 seconds.
- K. Titrate sample to pH 4.5. This must be done slowly so as not to miss the exact pH. Record titrant volume.
- L. The minimum titrant volume to be employed using high titrant is 1 mL. If high titrant is being used, go to low titrant; if low titrant doesn't work, use more sample. Be aware of sample volume that may be needed for other analyses. Do not dilute.
- M. When titrating the sample, be sure to allow time for the pH to equilibrate so that the inflection point will not be passed.
- N. Place pH probe in 7 buffer between samples. If this does not read 7, recalibrate between 4 and 7.
- O. Potentiometric titration of low alkalinity
  - a. For alkalinity of < 20 mg/L titrate 100 – 200 mL as above using a 10 mL microburet and 0.02 N acid solution.

- b. Stop titration at pH in range of 4.3-4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume. See note below.

Note : For method 2320B, if the pH of the sample is above 8.3, the sample needs to be titrated for phenolphthalein alkalinity, first check original pH. If it is not above 8.3, the phenolphthalein result will be below the minimum detection limit. If the original pH is above 8.3, titrate sample as in above procedure but down to 8.3 instead of 4.5, and record titrant volume in box A on the alkalinity bench sheet. Then proceed with the regular procedure titrating to pH 4.5, and recording this result in box B on the alkalinity bench sheet, then carefully titrate exactly 0.3 pH units lower to pH 4.2 and record titrant volume at this level, in box C on the alkalinity bench sheet.

- P. Potentiometric titration of high alkalinity: Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp endpoint.
  1. For >1000 mg CaCO<sub>3</sub>/L use 0.1 N titrant
    - i. For alkalinity of > 1000 mg CaCO<sub>3</sub>/L, titrate 25 – 50 mL as above using a 50 mL burette and 0.10 N acid solution.
    - ii. Stop titration at pH in range of 4.5, record volume and exact pH. See note above.

## IX. CALCULATIONS

The detection limit is 1.0 mg/L CaCO<sub>3</sub>.

Potentiometric titration to pH 4.5 (high alkalinity)

$$\text{Total Alkalinity mg/ L CaCO}_3 = \frac{A \times N \times 50,000}{\text{mL of Sample}}$$

Where: A = mL Standard Acid to pH 4.5  
N = Normality Standard Acid

Potentiometric titration of low alkalinity = T below

Method 2320B calculations

Use the following notation in below calculations:

P = Phenolphthalein alkalinity

T = Total alkalinity

$$P = \frac{A \times N \times 50,000}{\text{mL of Sample}}$$

$$T = \frac{(2B-C) \times N \times 50,000}{\text{mL of Sample}}$$

mL of Sample		mL of Sample	
If P = 0	Carbonate = 0	Bicarbonate = T	
If P < 1/2T	Carbonate = 2P	Bicarbonate = T-2P	
If P = 1/2T	Carbonate = 2P	Bicarbonate = 0	
If P > 1/2T	Carbonate = 2(T-P)	Bicarbonate = 0	
If P = T	Carbonate = 0	Bicarbonate = 0	

Where: A = mL titrant to pH 8.3  
B = mL titrant to pH 4.5  
C = mL titrant to pH 4.2  
N = normality Standard Acid

## X. QUALITY CONTROL

- A. Run a laboratory control sample (LCS) for each batch of samples (maximum of 20 samples per day). If the LCS does not fall in the range of 80 to 120%, corrective action must be taken to find the problem and correct it.
- B. Run a preparation blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than the reporting limit.
- C. Analyze a sample duplicate every 10 to 20 samples (depending on specific client requirements). Relative percent difference (RPD) on duplicates should be less than 20%.
- D. The Excel file for calculations is located in "V:\Wetchem\TESTS\CarbonDioxide Alkalinity\".
- E. Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

## **XI. CORRECTIVE ACTION**

- A. If the preparation blank is higher than the reporting limit, all samples less than ten times the concentration of the blank must be reanalyzed.
- B. If the laboratory control sample (LCS) is out of the range of 80 to 120%, and the % recovery is high (higher than 120%), only sample concentrations less than the reporting limit are acceptable data. Otherwise, all data with concentrations above the method detection limit must be reanalyzed with an LCS in the range of 80 to 120%. If the LCS is low (less than 80%), all samples must be reanalyzed.
- C. If the relative percent difference (RPD) between the sample and the sample duplicate are out of the range of 20%, the sample should be repeated one more time to make sure that the problem is not caused by analyst's error. If the RPD is still higher than 20% the sample is flagged on the final report with a "\*".

## **XII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **XIII. SAFETY**

- A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be worn when handling samples and chemicals.

## **XIV. METHOD PERFORMANCE**

- A. Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/L, CaCO <sub>3</sub>	Precision as Standard Deviation mg/L, CaCO <sub>3</sub>	Accuracy as	
		Bias, %	Bias, mg/L, CaCO <sub>3</sub>
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

## XV. REFERENCES

- A. Methods for the Chemical Analysis of Water and Wastes, EPA Series Method 310.1.
- B. Standard Methods for the Examination of Water and Wastewater, 14<sup>th</sup> Edition, p.278, method 403, (1975).
- C. Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, p. 2-26, method 2320B, (1992).

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**ORGANICS: SOP 201    REVISION #: 19    EFFECTIVE DATE: 041110**

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**GC/MS SEMIVOLATILES  
BY EPA METHOD 625 AND SW846 METHOD 8270C AND 8270D  
INCLUDING ADDITIONAL APPENDIX IX COMPOUNDS**

**APPROVALS:**

Lab Director: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Data Quality Manager: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

Section Supervisor: \_\_\_\_\_ Date: \_\_\_/\_\_\_/\_\_\_

## **Changes Summary**

### **Revision 19, 4/11/10**

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

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1. Identification of the Test Method
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## **1.0 Identification of the Test Method**

This SOP is based primarily on SW-846 Method 8000B/8000C/8270C/8270D. Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP.

## **2.0 Applicable Matrix or Matrices**

This SOP is used for the analysis of semi-volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.).

## **3.0 Detection Limit – Reporting Limits found in the Appendix**

## **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 the Appendix 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 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

## **5.0 Summary of the Test Method**

5.1 After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

## **6.0 Definitions –**

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

## **7.0 Interferences**

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

## **8.0 Safety**

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed 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 gloves and lab coats is highly recommended.
- 8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 8.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

## 9.0 Equipment & Supplies

- a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for splitless injection.
- b Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column.
- c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.
- d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.
- e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.
- f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- g Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified time or scan-number limits.
- h Micro syringes – gas tight 5µL and larger.
- i Liners – 2mm or 4mm single goose-neck.
- j Septa 11mm.
- k Seals- dual vespel stainless steel or gold plated 0.8mm.
- l Vials- 2ml and larger amber.
- m volumetric flasks- 10ml and larger class A with glass stopper.

## 10.0 Reagents and Standards –

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- Reagent grade chemicals shall be used in all tests unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other

- grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination
- 10.2 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.
- 10.3 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook.
- 10.3.1 The Decafluorotriphenylphosphine (DFTPP) tuning standard is prepared as follows (includes benzidine, pentachlorophenol and 4,4'-DDT): Using a 100 $\mu\text{L}$  syringe, 100 $\mu\text{L}$  (GCM-150, Ultra Scientific @ 1000 $\mu\text{g}/\text{mL}$ , or equivalent) is injected into a 2.0mL volumetric flask containing approximately 1.2mL methylene chloride (Trace Grade) and diluted to volume with same making a 50 $\mu\text{g}/\text{mL}$  standard. After capping and inverting several times, the solution is transferred into 2 labeled 2ml, teflon-lined, screw-capped vials and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months. A direct injection of 1.0 $\mu\text{L}$  is used to tune the instrument.
- 10.3.2 Calibration standards are prepared from a 200 $\mu\text{g}/\text{mL}$  working standard at a minimum of five concentrations. Calibration standards are prepared semi-annually unless the initial calibration verification standard indicates a problem. To makeup the 200 $\mu\text{g}/\text{mL}$  working standard inject the indicated amount of the following standards (or equivalent) into a 10mL volumetric containing approximately 5mL methylene chloride (Trace Grade) and dilute to volume with the same. After capping and inverting several times, the solution is transferred into an appropriate labeled vial, teflon-lined, screw-capped vial and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months. BNA intermediate standards are made @ 200ppm. Refer to Std prep in LIMS.

**Additional Appendix IX compounds can be added using 3 additional mixes, App. IX A,B,C. Refer to standards log in LIMS.**

To makeup the calibration standards, using a 1ml syringe add the appropriate amount of methylene chloride (trace grade) to a 2ml vial. Add the indicated amount of each intermediate standard to the vial. Add 20ul of internal standard to each screw-capped vial and stored in the freezer at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for up to 6 months. Refer to standards in LIMS.

The CCV is made at 50µg/mL. Occasionally, unusual compounds are added to the mix so it is best to check the BNA standards in LIMS for exact standard makeup. Note: MS list spikes and full list spikes for LCS and/or MS/MSD are prepared from an alternate source or lot number other than the calibration standards. Refer to standard logs in LIMS for details.

## 11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling. Soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

## 12.0 Quality Control

12.1 Internals - All samples and QC are spiked with internal standards.

12.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits for the surrogates. See section **Table 2** for criteria and corrective action. If any surrogate recoveries are below 10%, samples must be re-extracted if sample is available.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be re-extracted/reanalyzed for the failed analytes only or the data must be evaluated for flagging due to QC problems.

12.4 Method Blanks - The concentration of all method target analytes should be below the MDL for each method target analyte (**<RL for common lab contaminants and <1/2 RL /LOQ, <1/10regulatory limit or sample concentration for DOD QSM projects**). The first step of corrective action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, may need to be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank.

- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the full list spiking solution is used). Control limits for the MS/MSD recoveries are the same as those for the LCS found in the appendix. RPD limits are found on the LCS report forms in the appendix. Samples which do not meet these criteria due to matrix must be evaluated for flagging on the final report due to QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM , DOD limits will be used.**
- 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 two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 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.
- 12.5.5 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

### 13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

- 13.2 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed (only three concentration levels are required for *Federal Register* Method 625) and shown to meet the initial calibration criteria before any sample analysis may be performed. Method 625 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient  $r$  of  $\geq 0.995$  linear, 0.99 and six

points for quadratic. Method 8270C requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as  $r$  is  $\geq 0.995$  linear, 0.99 and six points for quadratic. In addition, there are calibration check compounds (CCCs) which must have a %RSD less than 30% and system performance check compounds (SPCCs) which must meet a minimum average response factor of 0.050. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve range from 1.0ug/mL to 100ug/mL. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason and dated with the quantitation report and chromatogram. Refer to SOP-QS07 for guidance. All integrations are checked for acceptability. Response factors of less than 0.050 must be supported by the mass spectrum of the lowest standard. Retention times are set using the midpoint of the curve. **No quadratic curves are used for South Carolina. For 8270D the RSD for each target analyte should be less than or equal to 20% and each calibration level should meet the minimum response factors listed in Table 2. If the 20% RSD is not met, then the minimum correlation coefficient for the curve must be 0.99. If more than 10% of the compounds do not meet the 20% RSD or minimum correlation coefficient of 0.99, then the chromatographic system is considered too reactive to begin analysis. Injector maintenance should be performed and repeat the calibration procedure.**

CCCs: <u>Base/Neutral</u>	<u>Acid</u>
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-phenylamine	Phenol
Di-n-octyl-phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

SPCCs: <u>Base/Neutral</u>	<u>Acid</u>
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

13.3 Initial Calibration Verification (ICV) - A second source standard at the 50  $\mu\text{g/mL}$  level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125% (**70-130% for 8270D & 80-120% for DOD4.1**). If the second source recovery is above 125% or 130% for 8270D, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 75% or 70% for 8270D, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. If any compound in the ICV exceeds the criteria above, it may be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis. Any manual integrations are documented by inclusion of the integrated

signals with the quantitation report and chromatogram. All integrations are checked for acceptability. For ICV standard preparation, refer to LIMS.

- 13.4 Continuing Calibration Verification (CCV)- Every 12 hours a CCV at 50 µg/mL must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for 8270C consists of the same SPCC requirements as the initial calibration. The CCCs must be less than or equal to 20% difference or drift (%D, calculations follow in section 7.9). If any of the CCCs do not meet the above limits, then all required analytes must be <20%D. Internal standard areas should be within 50 to 200 percent of the area of the curve midpoint or the previous CCV. Retention times for the internal standards should be within 30 seconds of the retention time of the curve midpoint or the previous CCV. Method 625 and DoD QSM requires a %D of less than 20% for all required analytes. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30%D, it must be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis. **For 8270D, the 20% difference criteria must be applied to all compounds. If the criterion is not met for more than 20% of the compounds included in the initial calibration, then the GC system should undergo maintenance. If this does not solve the problem, then the initial calibration should be repeated. Each of the most common target analytes should meet the minimum response factors listed in Table 2. In situations where the failed compound is present, the data must be flagged as estimated. If the compound fails high in the CCV and is not present, the result can be reported as non-detect.**
- 13.5 LCS - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS containing all regular full list calibrated compounds is spiked into deionized water or sodium sulfate for soil using an alternate source or lot number than the calibration standards. See the LCS report forms in the appendix for example laboratory generated limits and the NPDES limits for 625 samples. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. If enough data points are not present to generate limits, the limits default to CLP spike limits for spike analytes or 10-100% for all other analytes.  
**When analyzing samples for DOD QSM , DOD limits will be used.**
- 13.6 See SOP QS05 for guidance.

#### 14.0 Procedure

Prior to using Federal Register 625, SW-846 8270C/8270D, or CLP (semivolatile method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3540, 3550, 3580, EPA method 625 or CLP).

- 14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.

14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (every 24 hours for *Federal Register* Method 625 except for South Carolina which only allows 12 hours). The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port and must meet the following criteria. Degradation of DDT to DDE and DDE should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible for 8270C. **Tailing factor should be 2.0 for 8270D for benzidine and pentachlorophenol.** For NPDES samples, the benzidine (base/neutral) tailing factor must not exceed 3.0 while the pentachlorophenol (acid) tailing factor cannot exceed 5.0. The calculation for tailing factors is best illustrated in Figure 13 of the *Federal Register* Method 625 which has been placed in the appendix. If degradation is excessive and/or poor chromatography is seen, the injection port may require cleaning and maintenance. It may also be necessary to break off 15-30cm or more of the capillary column. The mass spectrum of DFTPP is acquired as follows: by using one scan at the apex peak, or by using the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or using the average across the entire peak. Background subtraction is accomplished using a single scan or more than 20 scans prior to the elution of DFTPP.

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

8270D has different tuning criteria for meeting DFTPP.  
See page 44 Table 3 of Method 8270D for criteria.

- 14.3 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch up to 20 samples.
- 14.4 Samples - Prior to analysis, 1.0 mL samples are prepared by verifying volume and spiking with 20uL of the internal standard solution.
- 14.5 Instrument sequence-The instrument sequence log is filled out prior to sample analyses.

An example of a typical instrument sequence log follows:

1-SEQ-TUN1 (12:00 am)

2-SEQ-CCV1

- 3-SEQ-BS1
- 4-SEQ-BLK1
- 5-Sample
- 6-Sample
- 7-Sample
- 8-Sample
- 9-Sample
- 10-Sample
- 11-Sample
- 12-Sample
- 13-Sample
- 14-SEQ-MS1
- 15-SEQ-MSD1
- 16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
- 17-SEQ-CCV2
- 18-Sample
- 19-Sample
- 20-Sample

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). **See SOP QS07 for guidance on manual integrations.**

14.6.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the curve midpoint. Retention time should be within 30 seconds of the retention time of the curve midpoint. If not, the sample and historical data should be evaluated to determine the cause of the problem. If matrix effect is confirmed by re-extraction/reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity.

14.6.2 Surrogates – Control limits are determined annually by charting LCSs and method blanks. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. All of the three surrogates for each fraction must be within the control limits in order for the extraction batch to be in control. If a surrogate exceeds the limits, the reason for the malfunction must be determined and a corrective action report must be completed. The sample must be reanalyzed, re-extracted or flagged for QC problems. *Federal Register* Method 625 contains no criteria for surrogate recovery. **When analyzing samples for DOD QSM , DOD limits will be used.**

Surrogate	Water	Soil/Sediment
Nitrobenzene-d5	40-110	35-100

2-Fluorobiphenyl	50-110	45-105
Terphenyl-d14	50-135	30-125
Phenol-d6	30-110	40-100
2-Fluorophenol	20-110	35-105
2,4,6-Tribromophenol	40-125	35-125

- 14.6.3 Analyte concentration must be within the range of the calibration curve after rounding to 3 significant figures. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.
- 14.6.4 The qualitative identification of compounds is based on retention time and a comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum from the NBS database (NBS75K.I). This database is used as it contains relatively uncontaminated mass spectra of each target compound which cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes. Characteristic ions from the reference mass spectrum library are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. In addition, the following criteria must be met. The RRT of the sample analyte must be within 0.06 RRT units of the RRT of the standard analyte. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of the same ions in the reference spectrum. Structural isomers that produce very similar mass spectra should be identified as individual isomers so long as their GC retention times differ substantially. A library search may be made for analytes not associated with the calibration for the purpose of tentative identification. NOTE: The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.
- 14.6.5 Quantitation - Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response factor is 15%(20% 8270D) or less, then the concentration may be determined using the average response factor ( $\overline{RF}$ ) from the initial calibration data. Otherwise, the concentration must be determined from equations based on internal standard calibration using either linear or non-linear calibration. Calculations follow in Section 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.

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

- 15.2 The RF is calculated as follows:  
where:

$A_s$  = Peak area (or height) of the analyte or surrogate.

$A_{is}$  = Peak area (or height) of the internal standard.

$C_s$  = Concentration of the analyte or surrogate.

$C_{is}$  = Concentration of the internal standard.

- 15.2.1 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within  $\pm 20\%$  for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place. **20% difference for 8270D and DOD QSM.**

- 15.2.2 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to  $\mu\text{g/L}$ .]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

$A_s$  = Area (or height) of the peak for the analyte in the sample.

$A_{is}$  = Area (or height) of the peak for the internal standard.

$C_{is}$  = Concentration of the internal standard in the volume extracted in  $\mu\text{g/L}$ .

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

$V_i$  = Volume of the extract injected ( $\mu\text{L}$ ). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$  = Mean response factor from the initial calibration.

$V_s$  = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

*The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.*

- 15.2.3 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to  $\mu\text{g}/\text{kg}$ .]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_s)(\overline{C_{is}})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where:  $A_s$ ,

$A_{is}$ ,  $C_{is}$ ,  $D$ , and  $\overline{RF}$  are the same as for aqueous samples, and

$W_s$  = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

*The 1000 in the denominator represents the number of  $\mu\text{L}$  in 1 mL. If the injection ( $V_i$ ) is expressed in mL, then the 1000 may be omitted.*

- 15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, and/or Technical Director or Quality Assurance Manager.

## 16.0 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 is completed by each analyst and then provided to the supervisor or QAO for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM, DOD limits will be used.** See method 8270C/8270D/625 for method performance.

## 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**

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**

*40 CFR, Part 136; Appendix A*  
*Test Methods for Evaluating Solid Waste, SW-846*  
*National Environmental Laboratory Accreditation Conference; CH. 5, 2003*  
*USACE, EM 200-1-3; Appendix 1; Shell, 2/2001*  
*DOD Quality Systems Manual for Environmental Laboratories,*

## **22.0 Tables, Diagrams, Flowcharts and Validation Data**

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).  
22.2 Table 2, QA/QC summary table  
22.3 Table 3, Technical Completeness / Accuracy Checklist  
22.4 Table 4, Data Reviewers Checklist(s)  
22.5 Table 5, 625 QC Limits  
22.6 Table 6, Standards Used  
22.7 Table 7, Internal Standard Associations  
22.8 Figure 13 from *Federal Register* method 625

**TABLE 1**

Analyte (Water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3'-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L

<b>Analyte (Water)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
<b>Analyte (Soil)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3'-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg
Benzo (a) pyrene	83.3	167	333	ug/Kg

<b>Analyte (Soil)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg
<b>Analyte Low PAH (Water)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1-Methylnaphthalene	0.0250	0.0500	0.100	ug/L
2-Methylnaphthalene	0.0250	0.0500	0.100	ug/L
Acenaphthene	0.0250	0.0500	0.100	ug/L
Acenaphthylene	0.0250	0.0500	0.100	ug/L
Anthracene	0.0250	0.0500	0.100	ug/L
Benzo (a) anthracene	0.0250	0.0500	0.100	ug/L
Benzo (a) pyrene	0.0250	0.0500	0.100	ug/L
Benzo (b) fluoranthene	0.0250	0.0500	0.100	ug/L
Benzo (g,h,i) perylene	0.0250	0.0500	0.100	ug/L
Benzo (k) fluoranthene	0.0250	0.0500	0.100	ug/L
Chrysene	0.0250	0.0500	0.100	ug/L
Dibenz (a,h) anthracene	0.0250	0.0500	0.100	ug/L
Fluoranthene	0.0250	0.0500	0.100	ug/L
Fluorene	0.0250	0.0500	0.100	ug/L
Indeno (1,2,3-cd) pyrene	0.0250	0.0500	0.100	ug/L
Naphthalene	0.0250	0.0500	0.100	ug/L
Phenanthrene	0.0250	0.0500	0.100	ug/L
Pyrene	0.0250	0.0500	0.100	ug/L
<b>Analyte Low PAH (Soil)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg

Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
<b>Analyte (TCLP)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L



**Table 2 - Method Quality Control Requirements Summary**

<b>QC Check</b>	<b>Minimum Frequency / Requirements</b>	<b>Acceptance Criteria</b>	<b>Corrective Action for Failures / Data Useability</b>
Tune	At the beginning of sequence and every 12 hours	See section 14.2 for criteria.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
Calibration Curve	<ul style="list-style-type: none"> <li>Prior to analyzing any samples</li> <li>A minimum of 5-points for linear fits</li> <li>A minimum of 6-points for quadratic fits</li> <li>Low standard at or below the RL/LOQ level</li> </ul>	<ul style="list-style-type: none"> <li>For Linear or Quadratic calibration fits a RF of 0.995</li> <li>Average RSD for CCCs <math>\leq 30\%</math>, to use avg. RF <math>\leq 15\%</math>, Min. RF for SPCCs per method</li> <li>Manual integrations on curve standards must have supervisory approval</li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-evaluate curve mix and makeup</li> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> <li>Re-prepare the curve standards</li> </ul> <p>Samples cannot be analyzed until there is a passing calibration</p>
ICV	Alternate source standard to be analyzed after every calibration curve	80-120% for DOD QSM 4.1 75-125% for 8270C, 70-130% for 8270D	<ul style="list-style-type: none"> <li>Re-analyze an ICV from a different source</li> <li>Re-prepare and re-analyze the ICV</li> <li>Re-calibrate and verify standard preps and sources</li> </ul>
CCV	<ul style="list-style-type: none"> <li>At the beginning of every sequence</li> <li>Every 12 hours</li> </ul>	See section 13.4 for criteria.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MB	One per prep batch	<ul style="list-style-type: none"> <li>Must be <math>&lt; \frac{1}{2}</math> the RL/LOQ</li> <li><math>&lt; 1/10</math> regulatory limit</li> <li><math>&lt; 1/10</math> sample concentration</li> </ul>	<ul style="list-style-type: none"> <li>Re-analysis to confirm the positive value</li> <li>Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers</li> <li>If results are between the LOD and RL/LOQ, then assess the data and notify the PM for further action</li> <li>Re-prepare of samples associated with the MB</li> <li>NCR will be required for data reported</li> <li>Final Report data flagging will be required</li> </ul>
LCS	One per prep batch	DOD QSM, QAPP or client specified	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
LCSD	One per prep batch, when MS/MSD not included.	DOD QSM, QAPP or client specified	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MS	One per prep batch, if sample volume available.	DOD QSM, QAPP or client specified	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MSD	One per prep batch, if sample volume available.	DOD QSM, QAPP or client specified	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>

**Table 2 - Method Quality Control Requirements Summary**

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Internal Standard	<ul style="list-style-type: none"> <li>A mix is used per sample post - prep</li> </ul>	<ul style="list-style-type: none"> <li>50 – 150 % of the IS from CCV</li> <li>(midpoint of ICAL used per DOD)</li> </ul>	<ul style="list-style-type: none"> <li>If holding time is expired, fill out a NCR and follow directions from PM</li> <li>Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution</li> <li>Re-injection or analysis</li> <li>Re-prep followed by re-analysis</li> <li>Follow guidelines from SOP QS05</li> </ul>
Surrogates	<ul style="list-style-type: none"> <li>A mix is used per sample prior to sample prep</li> </ul>	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>If holding time is expired, fill out a NCR and follow directions from PM</li> <li>Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution</li> <li>Re-injection or analysis</li> <li>Re-prep followed by re-analysis</li> <li>Follow guidelines from SOP QS05</li> </ul>
DOC Study	<ul style="list-style-type: none"> <li>Initially per analyst prior to reporting data</li> <li>Annually</li> <li>Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>Must meet the criteria of the LCS for average accuracy</li> </ul>	<ul style="list-style-type: none"> <li>Re-prep and /or re-analysis</li> </ul>
MDL Study	Once per year	<ul style="list-style-type: none"> <li>Calculated value must be greater than 10% of the Spike Level</li> <li>Calculated value must be less than the Spike level</li> </ul>	<ul style="list-style-type: none"> <li>Re-prep and /or re-analysis</li> <li>Follow guidelines from SOP QS05</li> </ul>
LOD Verification	Every quarter	<ul style="list-style-type: none"> <li>Parameter must be detected</li> <li>Check for Ion Abundance on MS methods</li> <li>the response must be 3-times the noise level</li> </ul>	<ul style="list-style-type: none"> <li>Re-prep and /or re-analysis</li> <li>Follow guidelines from SOP QS05</li> </ul>
LOQ Verification	Every quarter	<ul style="list-style-type: none"> <li>Bias Requirement: Inorganics 50-150% Organics 10-150%</li> <li>The LOQ value must be greater than the LOD value</li> </ul>	<ul style="list-style-type: none"> <li>Re-prep and /or re-analysis</li> <li>Follow guidelines from SOP QS05</li> </ul>

### **Table 3, Technical Completeness / Accuracy Checklist**

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

### **Table 4, Data Reviewers Checklist (Prior to approving data)**

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

## ANALYST DATA REVIEW CHECKLIST

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method:</b> <b>624/8260B/8270C/625 (Circle One)</b>

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?				
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.( eg. m/p-xylene, ketones,etc.).				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs,SPCCs and/or 20%D for all analytes.				
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?				
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?				
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?				
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?				

Comments on any "No" response:

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Primary-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

Table 5 - 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Table 6 - BNA STANDARDS USED

<u>base/neutral mix (2000ppm)</u>	<u>acids mix (2000ppm)</u>
bis(2-Chloroethyl)ether	2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether	2-Methylphenol
1,3-Dichlorobenzene	4-Methylphenol
1,2-Dichlorobenzene	Benzoic acid
1,4-Dichlorobenzene	4,6-Dinitro-2-methylphenol
Hexachloroethane	4-Nitrophenol
N-Nitroso-di-methylamine	2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine	2,4,6-Trichlorophenol
2,4-Dinitrotoluene	Phenol
2,6-Dinitrotoluene	Pentachlorophenol
Fluorene	2-Nitrophenol
Dimethylphthalate	4-Chloro-3-methylphenol
Hexachlorocyclopentadiene	2,4-Dichlorophenol
Anthracene	2,4-Dimethylphenol
4-Bromophenyl-phenylether	Benzoic acid
Di-n-butylphthalate	
bis(2-Chloroethoxy)methane	
1,2-Diphenylhydrazine	<u>semivoa misc.mix(2000ppm)</u>
Fluoranthene	Aniline
Hexachlorobenzene	Benzyl alcohol
N-Nitrosodiphenylamine	Carbazole
Phenanthrene	4-Chloroaniline
Hexachlorobutadiene	Dibenzofuran
Isophorone	2-Methylnaphthalene
Naphthalene	2-Nitroaniline
Nitrobenzene	3-Nitroaniline
1,2,4-Trichlorobenzene	4-Nitroaniline
Acenaphthene	Pyridine
Acenaphthylene	
2-Chloronaphthalene	<u>Benzidine mix (2000ppm)</u>
4-Chlorophenyl-phenylether	Benzidine
Diethylphthalate	3,3'-Dichlorobenzidine
Benzo(a)anthracene	
Bis(2-ethylhexyl)phthalate	
Butylbenzylphthalate	
Chrysene	<u>Individual or misc. mixes (2000/5000/20,000ppm)</u>
p-(Dimethylamino)azobenzene	Caprolactam
Pyrene	Benzaldehyde
Benzo(b)fluoranthene	Atrazine
Benzo(k)fluoranthene	1,1'-Biphenyl
Benzo(g,h,i)perylene	1,4-Dioxane
Benzo(a)pyrene	1-methylnaphthalene
Dibenz(a,h)anthracene	2,6-dichlorophenol
Di-n-octylphthalate	2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene	

<u>BNA internals (2000ppm)</u>	<u>Acid surrogate (7500ppm)</u>
1,4-Dichlorobenzene-d4 (I.S)(1)	2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)	Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)	2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)	2,-Chlorophenol-d4 (S)
Chrysene-d12 (I.S) (92))	<u>BN surrogate (5000ppm)</u>
Perylene-d12 (I.S) (101)	Nitrobenzene-d5 (S)
	Terphenyl-d14 (S)
	2-Fluorobiphenyl (S)
	1,2-Dichlorobenzene-d4 (S)

**Table 7 INTERNAL STANDARD ASSOCIATION /  
QUANT MASS**

COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 (I.S) (79)		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 (I.S)(35)		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 (I.S) (92)		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 (I.S) (59)		164	Perylene-d12 (I.S) (101)		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276

**I.S=internal standard, Q.M=quant mass, S=surrogate**

**Table 7 INTERNAL STANDARD ASSOCIATION /  
QUANT MASS (Continued)**

INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE					
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
0-Toluidine	1	106		59	
	1		Phenanthrene-d10 (I.S) (79)		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 (I.S)(35)		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 (I.S) (92)		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12- Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 (I.S) (59)		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 (I.S) (101)	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	

I.S=internal standard, Q.M=quant mass, S=surrogate

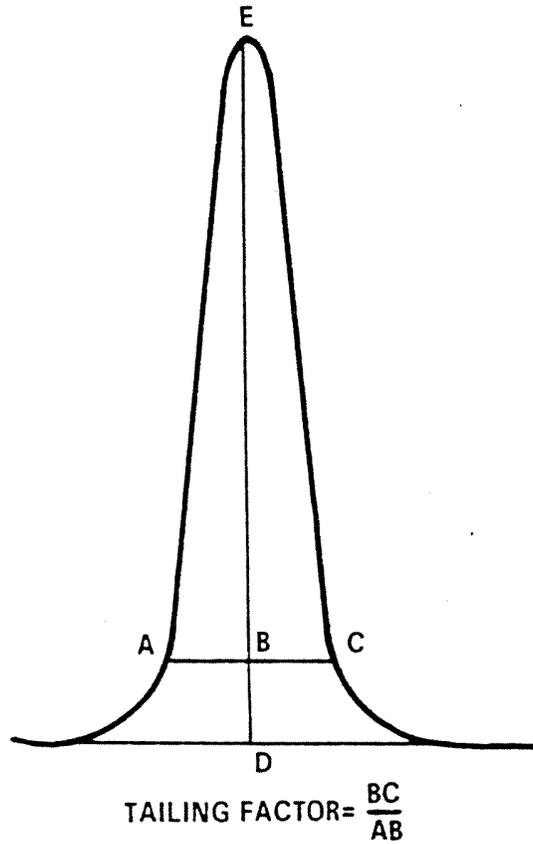
Analyte (water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3'-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L

<b>Analyte (Water cont'd)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
<b>Analyte (Soil)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3'-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg

Analyte (Soil Cont'd)	DL	LOD	MRL/LOQ	Units
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg
Benzo (a) pyrene	83.3	167	333	ug/Kg
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg

<b>Analyte (Low PAH Water)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1-Methylnaphthalene	0.0250	0.0500	0.100	ug/L
2-Methylnaphthalene	0.0250	0.0500	0.100	ug/L
Acenaphthene	0.0250	0.0500	0.100	ug/L
Acenaphthylene	0.0250	0.0500	0.100	ug/L
Anthracene	0.0250	0.0500	0.100	ug/L
Benzo (a) anthracene	0.0250	0.0500	0.100	ug/L
Benzo (a) pyrene	0.0250	0.0500	0.100	ug/L
Benzo (b) fluoranthene	0.0250	0.0500	0.100	ug/L
Benzo (g,h,i) perylene	0.0250	0.0500	0.100	ug/L
Benzo (k) fluoranthene	0.0250	0.0500	0.100	ug/L
Chrysene	0.0250	0.0500	0.100	ug/L
Dibenz (a,h) anthracene	0.0250	0.0500	0.100	ug/L
Fluoranthene	0.0250	0.0500	0.100	ug/L
Fluorene	0.0250	0.0500	0.100	ug/L
Indeno (1,2,3-cd) pyrene	0.0250	0.0500	0.100	ug/L
Naphthalene	0.0250	0.0500	0.100	ug/L
Phenanthrene	0.0250	0.0500	0.100	ug/L
Pyrene	0.0250	0.0500	0.100	ug/L
<b>Analyte (Low PAH soil)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg
Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
<b>Analyte (TCLP)</b>	<b>DL</b>	<b>LOD</b>	<b>MRL/LOQ</b>	<b>Units</b>
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L

Figure 13



Example calculation: Peak Height = DE = 100 mm  
10% Peak Height = BD = 10 mm  
Peak Width at 10% Peak Height = AC = 23 mm  
AB = 11 mm  
BC = 12 mm  
Therefore: Tailing Factor =  $\frac{12}{11} = 1.1$

Figure 13. Tailing factor calculation.

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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**ORGANICS: SOP 202**

**REVISION #: 22**

**EFFECTIVE DATE: 093009**

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**GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B  
INCLUDING APPENDIX IX COMPOUNDS**

**APPROVALS:**

Lab Director:



Date: 10/5/09

Data Quality Manager:



Date: 10/5/09

- Section Supervisor:



Date: 10/7/09

## **Changes Summary**

### **Revision 22, 9/30/09**

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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22. Tables, Diagrams, Flowcharts and Validation Data

## 1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

## 2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. **Detection Limit:** See **Table 1** of this SOP.

## 4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. 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.

## 5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

## 6. Definitions

6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

amu	atomic mass unit
BFB	Bromofluorobenzene

°C	degrees Centigrade
CLP	Contract Laboratory Program
DOD	Department of Defense
EICP	extracted ion current profile
G	gram or grams
GC/MS	Gas Chromatograph/Mass Spectrometer
I.D.	inner diameter
ISTD	internal standard
µm	micrometer
µL	microliter
mL	milliliter
mm	millimeter
ng	nanogram
P&T	purge and trap
SURR	surrogate
SPCC	System Performance Check Compound
TCLP	Toxicity Characteristic Leaching Procedure
USACE	United States Army Corps Of Engineers
VOA	volatile organic analysis

## 7. Interferences

- 7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

## 8. Safety

- 8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

## 9. Equipment & Supplies

- 9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.
- 9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.
- 9.3 M.S. : HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]
- 9.4 Purge and Trap Unit
- 9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

- 9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.
- 9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- 9.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.
- 9.7 Microsyringes – 1.0, 5.0, 10, 25, 100, 250, 500 and 1000  $\mu$ L.
- 9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.
- 9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.
- 9.10 Disposable pasteur pipets.
- 9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.
- 9.12 Spatula - stainless steel.
- 9.13 Glass scintillation vials - 20mL with screw caps.
- 9.14 Nitrile Gloves
- 9.15 pH paper (measures pH from 0-14).

## 10. Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.2 Organic-free reagent water - obtained from a modulab system.
- 10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)
- 10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)
- 10.5 Sodium bisulfate,  $\text{NaHSO}_4$  – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- 10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of  $-15^\circ\text{C} \pm 5^\circ\text{C}$  or less from the date they are received/prepared. The freezer

temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C or less for up to 6 months (**1 week for South Carolina samples**). A direct injection of 1µL (or equivalent purge) is used to tune the instrument.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor, Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. The internal standard/surrogate solution will be replaced if the -50%-200% criteria fails in the CCV when calculated against the previous CCV.

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the VOA standards log book for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard. See the appendix for analytes in the main mixes.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L (5µL purge) standard is made using 25µL of this standard to 50mL of reagent water.

Stock Standard(CCV)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE (Cat#30265)	20000	25	20	200
Vinyl Acetate (#3766)	5000	100	80	200
Ketones (cat#30006)	5000	100	80	200
Liquid mix (C-349H-07)	2000	100	100	100
Custom mix (CCS-1037)	5000	50	40	100
Gases (cat#30042)	2000	100	100	100
Acrolein/Acrylonitrile (CC2098.10)	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor, Lot) and diluted to volume with same to make a 100-500ng/μL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50μg/L ICV/LCS/Matrix Spike is made using 25μL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/μL)	Syringe(μL)	Amount(μL)	Final Conc (ng/μL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

## 11. Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. All water samples are stored in the BlueIce refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).

## 12. Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for criteria and corrective action.
- 12.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits. See section 14.5.2 of this SOP for criteria and corrective action. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**
- 12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). The recoveries are used to generate control limits. The limits are in-house generated matrix spike limits or client specified limits for matrix spike analytes and 70-130% (or client specified limits) recovery for waters or soils for all other analytes if limits have not been generated. Limits for 624 LCSs are taken from table 5 of method 624. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reanalyzed for the failed analytes only. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used. South Carolina limits are 70-130% except for poor purgers which are 60-140%.**
- 12.4 Method Blanks - The concentration for method target analytes must be < ½ the Reporting Limit (also defined as the Limit of Quantitation). The first step of corrective action is to assess the affect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. A method blank is analyzed every 12 hour tune.
- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. Criteria for the MS/MSD recoveries are the same as the LCS limits. Limits for the RPDs are 30% RPD for water and soil.. Samples that do not meet these criteria due to matrix must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the

failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

- 12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
- 12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.
- 12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

### 13. Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.
- 13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.  
  
NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.
- 13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (**every 24 hours for *Federal Register Method 624* except for South Carolina which only allows 12 hours**). The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with background subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

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

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate.** All calibration standard manual integrations must be approved by for acceptability.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** Method 624 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient (*r*) of  $\geq 0.995$ . Method 8260B requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as *r* is  $\geq 0.995$  linear or  $\geq 0.99$  quadratic. In addition, there are calibration check compounds (CCCs) listed below which must have a %RSD less than 30% and five system performance check compounds (SPCCs) which must meet the average response factor criteria listed below. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride
SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30

- 13.5.2 Initial Calibration Verification - A second source standard at the 50 µg/L (5mL purge) level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. **When analyzing samples for DOD QSM Version 4.1, DOD limits (80-120%) will be used.** If the second source recovery is above 125%, the main standard has probably deteriorated for that compound. That standard must be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard has probably deteriorated for that compound and must be replaced. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management
- 13.5.3 Continuing Calibration Verification (every 12 hours) - A midpoint calibration standard (generally 50 µg/L - 5mL purge) must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for method 8260B consists of the same SPCC criteria as above and  $\leq 20\%$  drift or difference (calculations given in section 7.10) for the CCCs as listed above. The internal standards must also be evaluated as listed below. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30% D, it must be evaluated following the guidelines outlined in SOP QS05. If deemed acceptable, the analyst may continue analysis. **When analyzing samples for DOD QSM Version 4.1, DOD acceptance criteria of  $\leq 20\%$  drift or difference for all analytes will be used.**
- NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

#### Internal standard checks

- 13.5.3.1 Retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- 13.5.3.2 Response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +

100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

## 14. Procedure

- 14.1 LCS - An LCS is analyzed every 12 hour tune. Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See section 12 above for criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**
- 14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See [Table 2](#) for criteria and corrective action.
- 14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:
  - 14.3.1 Load the vial into the Archon autosampler in the expected position.
  - 14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.
  - 14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a corrective action report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

#### 14.4 Instrument sequence

**An example of a typical instrument sequence log follows:**

- 1-BFB Tune (12:00 am)
- 2-CCV
- 3-LCS
- 4-Method Blank
- 5-Sample
- 6-Sample
- 7-Sample
- 8-Sample
- 9-Sample
- 10-Sample
- 11-Sample
- 12-Sample
- 13-Sample
- 14-Sample
- 15-Sample
- 16-Sample
- 17-Sample MS
- 18-Sample MSD
- 19-BFB (12:00pm - 12 hours since last BFB/CCV)
- 20-CCV
- 21-LCS
- 22-Method Blank
- 23-Sample
- 24-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the continuing calibration. Retention time should be within 30 seconds of the retention time of the continuing calibration. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity. If not, the sample and historical data should be evaluated to determine the cause of the problem. Reanalysis is expected if it appears to be from a leak. If matrix effect is confirmed by reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect.

14.5.2 Surrogates – Control limits are determined by charting LCSs and method blanks. All of the surrogates must be within these limits in order for the analysis to be in control. If not, the reason for the malfunction must be determined and reanalysis may be necessary. If historical data indicates matrix, the sample would be flagged appropriately. When the surrogates exceed either the control limits, a corrective action report must be completed.

**Federal Register Method 624 contains no criteria for surrogate recovery. When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

Surrogate	WATER	SOIL/SEDIMENT
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed, the next sample must be checked for carryover and the sparge position must be checked for contamination through the analysis of a system blank. Any dilution should keep the concentration of the analyte in question within the mid-range of the curve.

14.5.4 Qualitative identification is made as indicated below.

14.5.4.1 The mass spectra are compared to reference spectra in a user-created data base especially compiled to contain relatively uncontaminated mass spectra of each target compound. Note: Such a file cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes.

14.5.4.2 The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

## 15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows: 
$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

$A_s$  = Peak area (or height) of the analyte or surrogate.

$A_{is}$  = Peak area (or height) of the internal standard.

$C_s$  = Concentration of the analyte or surrogate.

$C_{is}$  = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within  $\pm 20\%$  for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(V_s)(1000)}$$

where:

$A_s$  = Area (or height) of the peak for the analyte in the sample.

$A_{is}$  = Area (or height) of the peak for the internal standard.

$C_{is}$  = Concentration of the internal standard in the volume purged in ug/L.

$D$  = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made,  $D = 1$ . The dilution factor is always dimensionless.

$V_i$  = For purge-and-trap analysis,  $V_i$  is not applicable and is set at 1.

$\overline{\text{RF}}$  = Mean response factor from the initial calibration.

$V_s$  = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(W_s)(1000)}$$

where:  $A_s$ ,  $A_{is}$ ,  $C_{is}$ ,  $D$ , and  $\overline{\text{RF}}$  are the same as for aqueous samples.

$W_s$  = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

## 16. Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See [Table 2](#) for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

## 17. Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

## **19. Contingencies for Handling out-of-control or unacceptable data**

19.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

## **20. Waste Management.**

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

## **21. References**

- 21.1 40 CFR, Part 136; Appendix A
- 21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
- 21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
- 21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
- 21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005
- 21.6 DOD Quality Systems Manual for Environmental Laboratories version 4.1, 4/2009

## **22. Tables, Diagrams, Flowcharts and Validation Data**

**TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard**

<b>Parameter</b>	<b>RL Water ug/L</b>	<b>LowCal Water ug/L</b>	<b>RL Soil ug/KG</b>	<b>LowCal Soil ug/KG</b>
1,1,1 Trichloroethane	1.0	1.0	5.0	2.0
1,1,1,2-Tetrachlorethane	1.0	1.0	5.0	2.0
1,1,2,2-Tetrachloroethane	1.0	1.0	5.0	2.0
1,1,2-Trichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethene	1.0	1.0	5.0	2.0
1,2,4 Trichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dibromo-3-chloropropane	1.0	1.0	5.0	2.0
1,2-Dibromoethane	1.0	1.0	5.0	2.0
1,2-Dichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dichloroethane	1.0	1.0	5.0	2.0
1,2-Dichloropropane	1.0	1.0	5.0	2.0
1,3-Dichlorobenzene	1.0	1.0	5.0	2.0
1,4-Dichlorobenzene	1.0	1.0	5.0	2.0
2-Butanone	10	2.0	50	4.0
2-Hexanone	5.0	2.0	10	4.0
4-Methyl-2-pentanone	5.0	2.0	10	4.0
Acetone	10	2.0	50	4.0
Benzene	1.0	1.0	5.0	2.0
Bromochloromethane	1.0	1.0	5.0	2.0
Bromodichloromethane	1.0	1.0	5.0	2.0
Bromoform	1.0	1.0	5.0	2.0
Bromomethane	2.0	1.0	10	2.0
Carbon disulfide	1.0	1.0	5.0	2.0
Carbon tetrachloride	1.0	1.0	5.0	2.0
Chlorobenzene	1.0	1.0	5.0	2.0
Chloroethane	2.0	1.0	10	2.0
Chloroform	1.0	1.0	5.0	2.0
Chloromethane	2.0	1.0	10	2.0
Cis-1,2-Dichloroethene	1.0	1.0	5.0	2.0
Cis-1,3-Dichloropropene	1.0	1.0	5.0	2.0
Dibromochloromethane	1.0	1.0	5.0	2.0
Dibromomethane	1.0	1.0	5.0	2.0
Dichlorodifluoromethane	2.0	1.0	10	2.0
Ethylbenzene	1.0	1.0	5.0	2.0
Methylene chloride	2.0	1.0	10	2.0
M,p-Xylene	1.0	2.0	5.0	4.0
o-Xylene	1.0	1.0	5.0	2.0
Styrene	1.0	1.0	5.0	2.0

**TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard**

<b>Parameter</b>	<b>RL Water ug/L</b>	<b>LowCal Water ug/L</b>	<b>RL Soil ug/KG</b>	<b>LowCal Soil ug/KG</b>
Tetrachloroethene	1.0	1.0	5.0	2.0
Toluene	1.0	1.0	5.0	2.0
Trans-1-2 Dichlorethene	1.0	1.0	5.0	2.0
Trans-1-3-Dichloropropene	1.0	1.0	5.0	2.0
Trichloroethene	1.0	1.0	5.0	2.0
Trichlorofluroromethane	2.0	1.0	10	4.0
Vinyl chloride	2.0	1.0	10	4.0
MTBE	1.0	1.0	5.0	2.0
Naphthalene	1.0	1.0	5.0	2.0

**Table 2 - Method Quality Control Requirements Summary**

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Tune	At the beginning of sequence and every 12 hours	See section 13.4 for criteria.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
Calibration Curve	<ul style="list-style-type: none"> <li>Prior to analyzing any samples</li> <li>A minimum of 5-points for linear fits</li> <li>A minimum of 6-points for quadratic fits</li> <li>Low standard at or below the RL/LOQ level</li> </ul>	<ul style="list-style-type: none"> <li>For Linear or Quadratic calibration fits a RF of 0.995</li> <li>Average RSD for CCCs <math>\leq 30\%</math>, to use avg. RF <math>\leq 15\%</math>, Min. RF for SPCCs per method</li> <li>Manual integrations on curve standards must have supervisory approval</li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-evaluate curve mix and makeup</li> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> <li>Re-prepare the curve standards</li> </ul> <p>Samples cannot be analyzed until there is a passing calibration</p>
ICV	Alternate source standard to be analyzed after every calibration curve	75-125% for 8260B, 80-120% for DOD QSM 4.1	<ul style="list-style-type: none"> <li>Re-analyze an ICV from a different source</li> <li>Re-prepare and re-analyze the ICV</li> <li>Re-calibrate and verify standard preps and sources</li> </ul>
CCV	<ul style="list-style-type: none"> <li>At the beginning of every sequence</li> <li>Every 12 hours</li> </ul>	See section 13.5.3 for criteria.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
MB	One per prep batch	<ul style="list-style-type: none"> <li>Must be <math>&lt; \frac{1}{2}</math> the RL/LOQ</li> </ul>	<ul style="list-style-type: none"> <li>Re-analysis to confirm the positive value</li> <li>Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers</li> <li>If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action</li> <li>Re-prepare of samples associated with the MB</li> <li>NCR will be required for data reported</li> <li>Final Report data flagging will be required</li> </ul>
LCS	One per prep batch	Most stringent criteria listed within the LIMS.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>
LCSD	One per prep batch, when MS/MSD not included.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>Follow guidelines from SOP QS05</li> </ul>

**Table 2 - Method Quality Control Requirements Summary**

<b>QC Check</b>	<b>Minimum Frequency / Requirements</b>	<b>Acceptance Criteria</b>	<b>Corrective Action for Failures / Data Useability</b>
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>• Follow guidelines from SOP QS05</li> </ul>
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>• Follow guidelines from SOP QS05</li> </ul>
Internal Standard	<ul style="list-style-type: none"> <li>• A mix is used per sample post - prep</li> </ul>	<ul style="list-style-type: none"> <li>• 50 – 150 % of the IS from CCV</li> </ul>	<ul style="list-style-type: none"> <li>• If holding time is expired, fill out a NCR and follow directions from PM</li> <li>• Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution</li> <li>• Re-injection or analysis</li> <li>• Re-prep followed by re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
Surrogates	<ul style="list-style-type: none"> <li>• A mix is used per sample prior to sample prep</li> </ul>	Criteria listed within LIMS or specified by client.	<ul style="list-style-type: none"> <li>• If holding time is expired, fill out a NCR and follow directions from PM</li> <li>• Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution</li> <li>• Re-injection or analysis</li> <li>• Re-prep followed by re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
DOC Study	<ul style="list-style-type: none"> <li>• Initially per analyst prior to reporting data</li> <li>• Annually</li> <li>• Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>• Must meet the criteria of the LCS for average accuracy</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and /or re-analysis</li> </ul>
MDL Study	Once per year	<ul style="list-style-type: none"> <li>○ Calculated value must be greater than 10% of the Spike Level</li> <li>○ Calculated value must be less than the Spike level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and /or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>
LOD Verification	Every quarter	<ul style="list-style-type: none"> <li>○ Parameter must be detected</li> <li>○ Check for Ion Abundance on MS methods</li> <li>○ the response must be 3-times the noise level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and /or re-analysis</li> <li>• Follow guidelines from SOP QS05</li> </ul>

**Table 2 - Method Quality Control Requirements Summary**

<b>QC Check</b>	<b>Minimum Frequency / Requirements</b>	<b>Acceptance Criteria</b>	<b>Corrective Action for Failures / Data Useability</b>
LOQ Verification	Every quarter	<ul style="list-style-type: none"> <li>○ Bias Requirement: Inorganics 50-150% Organics 10-150%</li>   <li>○ The LOQ value must be greater than the LOD value</li> </ul>	<ul style="list-style-type: none"> <li>● Re-prep and /or re-analysis</li> <li>● Follow guidelines from SOP QS05</li> </ul>

### **Table 3, Technical Completeness / Accuracy Checklist**

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

### **Table 4, Data Reviewers Checklist (Prior to approving data)**

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

**TOTAL ORGANIC CARBON  
(TOC)**

**SM5310C,  
SW846 METHOD 9060/9060A  
AND LLOYD KAHN METHOD**

**SOP NUMBER:** SOP-221

**REVISION NUMBER:** 8

**APPROVED BY:** *Betty DeVill*  
**SECTION MANAGER**

*Pandy D. Ward*  
**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** 04/28/09

**DATE OF LAST REVIEW:** 04/28/09

**TOTAL ORGANIC CARBON (TOC)**  
**BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN**  
METHOD “*DETERMINATION OF TOC IN SEDIMENT*”

**I. SCOPE AND APPLICATION**

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060/9060A and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the Lloyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices. SW846 Method 9060/9060A and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

**II. SUMMARY OF METHOD**

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO<sub>2</sub> formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

**III. SAMPLING HANDLING AND PRESERVATION**

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory

indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

- 3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.
- 3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH  $\leq$  2) with HCl or H<sub>2</sub>SO<sub>4</sub>. Both water and soil samples are stored at 4°C.

#### **IV. INTERFERENCES**

##### **4.1 WATER METHOD**

- 4.1.1 Removal of carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.
- 4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

##### **4.2 SOIL METHOD**

- 4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.
- 4.2.2 The infrared detector is sensitized to CO<sub>2</sub> and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

## V. DEFINITIONS

- 5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.
- 5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.
- 5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.
- 5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.
- 5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.
- 5.7 LABORATORY CONTROL SAMPLE (LCS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).
- 5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.
- 5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory

performance using test materials that have been prepared independently from the normal preparation process.

- 5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

## VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C ( $\pm$  2°C).

- 6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

- 6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

- 6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

- 6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

### 6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL  
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL  
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL  
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL  
25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL  
50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL  
100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL

0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL  
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL  
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL  
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL  
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL  
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

#### 6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.
2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

- 6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

- 6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

- 6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

## VII. INSTRUMENTATION

- 7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

- 7.2 There is a Shimadzu autosampler which will hold 68 samples.

- 7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

## VIII. AQUEOUS SAMPLE PROCEDURE

- 8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.
- 8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**
- 8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.
1. 100 ppm
  2. 50 ppm
  3. 25 ppm
  4. 10 ppm
  5. 5.0 ppm
  6. 2.5 ppm
  7. 1.0 ppm
  8. Method blank
  9. LCS + 9 samples (including any sample QC
  10. 25 ppm
  11. 10 samples (including any sample QC)
  12. 50 ppm
- 8.8 Instrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

**TC Mode Instrument Settings:**

Analysis Temp: 900°C

Analysis Time: 6.5 minutes

Nitrogen Gas Flow: 60-100 psi (external regulator regulator)

**Oxygen Gas Flow: 40-60 psi (external regulator)**

**This is a step by step description of a routine soil TOC analysis.**

- 9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400 mL/minute and an oxygen flow of 180 mL/minute ( $\pm 3$  mL/minute).

**NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!**

- 9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.
- 9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).
- 9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.
- 9.5 If doing an initial calibration curve use an appropriate  $\mu$ L syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

$\mu$ L 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
50	15
100	30

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100  $\mu$ L of the 30% Sucrose standard with 500  $\mu$ L of water.

- 9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CC1+ date analyzed for ID) or Initial calibration – single analyses
  2. Method Blank(MB + date analyzed for ID) – single analyses
  3. LCS, 15 mg dextrose (LCS + date analyzed for ID) – single analyses
  4. NY Cert – 4 replicates
  5. Sample – 4 replicates
  6. Sample – 4 replicates
  7. Sample – 4 replicates
  8. Sample – 4 replicates
  9. Sample – 4 replicates
  10. CCV(CC1+ date analyzed for ID)2 – single analyses
  11. Sample – 4 replicates
  12. Sample – 4 replicates
  13. Sample – 4 replicates
  14. Sample – 4 replicates
  15. Sample – 4 replicates
  16. CCV (CC2+ date analyzed for ID) – single analyses
  17. Sample – 4 replicates
  18. Sample – 4 replicates
  19. Sample – 4 replicates
  20. Sample – 4 replicates
  21. Sample – 4 replicates
  22. CCV(CC3+ date analyzed for ID) – single analyses
  23. Sample – 4 replicates
  24. Sample – 4 replicates
  25. Sample – 4 replicates
  26. Sample – 4 replicates
  27. Sample – 4 replicates
  28. CCV(CC4+ date analyzed for ID) – single analyses
  29. SampleMS – 4 replicates
  30. SampleDUP – 4 replicates
  31. FCV(CC4+ date analyzed for ID) – single analyses
  32. FCB(FCB4+ date analyzed for ID) – single analyses
- 9.7 Samples should be stored away from light and at 4°C ( $\pm$  2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.
- 9.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are

exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot is taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.**

- 9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.
- 9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## X. QC REQUIREMENTS

- 10.1 Analyze a laboratory control sample (LCS) for each batch of samples **(maximum of 10 samples per day)**. If the LCS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.
- 10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.
- 10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.
- 10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are  $\leq 15\%$  for aqueous samples and  $\leq 20\%$  for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or  $\leq 30\%$  (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

- 10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.
- 10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than  $\pm$  the MDL.
- 10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.
- 10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

- 10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within  $\pm 10\%$ . A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060/9060A requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required number of sample replications.
- 10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

## **XI. CORRECTIVE ACTIONS**

### **11.1 INSTRUMENT RELATED**

1. ICV not within  $\pm 20\%$  (Soil) or  $\pm 10\%$  (SM 5310C0)
  - a. If the problem is with the solution.

- i. Re-prepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within  $\pm 30\%$  (Soil) or  $\pm 15\%$  (SM 5310C)
  - a. If the problem is with the solution.
    - i. Re-prepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalyzed.

**\* Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

#### **11.2 SAMPLE MATRIX RELATED**

1. Replicate analysis RPD not within  $\pm 20\%$  aqueous or  $\pm 50\%$  soil/sediment
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$  aqueous or  $\pm 50\%$  soil/sediment
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

## **XII. HEALTH AND SAFETY**

- A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.
- D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

## **XIII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

**XIV. METHOD PERFORMANCE**

**14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)**

<b>Characteristic Of Analysis Concentration determined, mg/L:</b>	<b>Spring Water</b>	<b>Spring Water +0.15 mg/L KHP*</b>	<b>Tap Water</b>	<b>Tap Water +10 mg/L KHP*</b>	<b>Municipal Wastewater Effluent</b>
<b>Replicate 1</b>	<b>0.402</b>	<b>0.559</b>	<b>2.47</b>	<b>11.70</b>	<b>5.88</b>
<b>Replicate 2</b>	<b>0.336</b>	<b>0.491</b>	<b>2.49</b>	<b>11.53</b>	<b>5.31</b>
<b>Replicate 3</b>	<b>0.340</b>	<b>0.505</b>	<b>2.47</b>	<b>11.70</b>	<b>5.21</b>
<b>Replicate 4</b>	<b>0.341</b>	<b>0.523</b>	<b>2.47</b>	<b>11.64</b>	<b>5.17</b>
<b>Replicate 5</b>	<b>0.355</b>	<b>0.542</b>	<b>2.46</b>	<b>11.55</b>	<b>5.10</b>
<b>Replicate 6</b>	<b>0.366</b>	<b>0.546</b>	<b>2.46</b>	<b>11.68</b>	<b>5.33</b>
<b>Replicate 7</b>	<b>0.361</b>	<b>0.548</b>	<b>2.42</b>	<b>11.55</b>	<b>5.35</b>
<b>Mean, mg/L</b>	<b>0.35</b>	<b>0.53</b>	<b>2.46</b>	<b>11.53</b>	<b>5.32</b>
<b>Std. Deviation: mg/L</b>	<b>0.02</b>	<b>0.03</b>	<b>0.02</b>	<b>0.21</b>	<b>0.23</b>
<b>%</b>	<b>6</b>	<b>6</b>	<b>1</b>	<b>2</b>	<b>4</b>
<b>Actual Value, mg/L</b>	<b>-</b>	<b>0.50</b>	<b>-</b>	<b>12.46</b>	<b>-</b>
<b>Recovery, %</b>	<b>-</b>	<b>106</b>	<b>-</b>	<b>93</b>	<b>-</b>
<b>Error, %</b>	<b>-</b>	<b>6</b>	<b>-</b>	<b>7</b>	<b>-</b>

\*KHP = potassium acid phthalate.

**14.2 There was no method performance data available for the soil procedure.**

**XV. REFERENCES**

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).
3. EPA SW-846, Method 9060/9060A.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

**APPENDIX I.**

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eighth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1" beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they are loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightning bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into correct position will take almost

- 3 hours to finish. Can view data as its coming off by selecting “view” “sample window”. After calibration is done review.
27. Select “File” then “New” then “sample run” “ok”
  28. General information screen: No change select “ok”
  29. Save as screen: Select today’s date for file name example 00month/00day/00year
  30. Select “save”
  31. Sample Table Screen: Select “insert” then select “ auto generate” enter
  32. **Page 1** sample group wizard sample source: select “calibration curve” then double click on box with 3 dots ...
  33. Open latest curve from calibration curves file
  34. Highlight latest curve and select “open”
  35. Should send you back to page 1 with calibration curve info submitted. Select “next”
  36. **Page 2** Sample Parameter: Enter final sample count for “number of samples” select “next”
  37. **Page 3** Calibration Curves: No changes Select “Next”
  38. **Page 4** Calibration Checks: No changes Select “Next”
  39. **Page 5** Controls: No changes select “finish”, Select “ok” on “Sparging/ Acid page.
  40. Type sequence as they are loaded on tray: ICV, ICB, LCSW, Sample #, client,etc.
  41. Once everything is typed in double check that it matches the way samples and QC are loaded..
  42. Click or select the lightening bolt symbol then select “use settings on PC”. Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
  43. Click or select “shut down Instructions”. Then select “standby” Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select “OK” if it looks the same.
  44. Start ASI measurement: External acid addition should have a check mark click on “start” analysis should begin to start.
  45. Click on view and chose “sample window” to watch curves come off and to see beginning values.

**Methane, Ethane, Ethene in Aqueous  
Samples by Modified RSK-175  
(Automated Headspace)**

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**SOP NUMBER:**

**SOP-236**

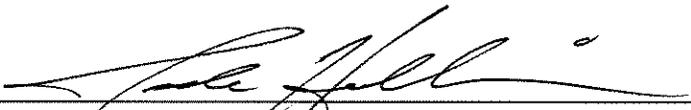
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**REVISION NUMBER:**

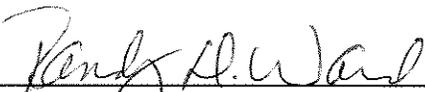
**1**

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**APPROVED BY:**

  
**SECTION MANAGER**

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**QUALITY ASSURANCE MANAGER**

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**EFFECTIVE DATE:**

**05/23/08**

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**DATE OF LAST REVIEW:**

**04/28/09**

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## **Methane, Ethane, Ethene in Aqueous Samples by Modified RSK-175 (Automated Headspace)**

### **I. SUMMARY**

The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples. Reporting limits for these are methane 2.0 ug/L, ethane 1.4 ug/L, and ethene 1.1 ug/L.

### **II. SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE**

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. Samples are collected in 40 ml VOA vials and shipped to the lab in coolers with ice. Water samples are stored in the Hobart in the sample storage room at a temperature of 4°C.

### **III. INTERFERENCES AND POTENTIAL PROBLEMS**

Methane found in the lab environment can be a source of contamination. The blank value is subtracted from the sample results.

### **IV. INSTRUMENTATION AND EQUIPMENT**

- A. Gas Chromatograph
  - 1. HP 5890 Series II (temperature programmable).
- B. Autosampler
  - 1. Tekmar 7000 Headspace autosampler
- C. Columns-Capillary columns.
  - 1. Carboxen 1006 PLOT column—30 meter x 0.53mm ID
- D. Data Acquisition and Processing Software.
  - 1. HP Chemstation system is interfaced to the HP-GC for data acquisition and storage.
  - 2. TARGET data system is interfaced to the acquisition systems. The system accepts, processes and stores acquired data.
- E. Glassware
  - 1. 25ml Graduated cylinder
  - 2. 20ml headspace vials with crimp tops( National Scientific)

3. Gastight syringes- 25, 50, 100, and 250uL

## V. STANDARDS

Gas standards are purchased from Restek and Supelco. The date they are received is noted on the container they are received in. The standards are given a sequential number the day they are opened and this is noted in the GC standards logbook. Standards for MEE are Scotty gases purchased in pressurized tanks. Calibration standards at a minimum of five levels are prepared by injecting the gas from a Scotty gas standard tank into capped 20 ml headspace vials with 15 ml of D.I. water using a gas-tight syringe. Usually 5,10,20,25,50,100,150,200ul and up to 5ml are used.

## VI. PROCEDURE

The following information describes the instrument and QC requirements to analyze the compounds that we do by this method.

### A. Instrumentation

#### 1. GC

- Initial Temperature: 35 ° C hold for 3.0 minutes.
- Ramp: 25 °C / minute to 225 ° C.
- Final Temperature 225 ° C hold for 3.08 minutes.
- Detector Temperature 230 ° C.

#### 2. Headspace Autosampler

- Platen: 80 °C./ Platen Equil.: 0.50 min.
- Sample Equil.: 2.0 min.
- Pressurization: 0.50 min./ Pressure Equil.: 0.25 min.
- Loop Equil.: 0.30 min.
- Injection Time: 1.0 min.
- Valve and Line Temp.: 95°C.

### B. Calibration and Quality Control

1. Refer to SW-846 Method 8000B for proper calibration techniques.
  - a. Five point minimum calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 percent over the working

range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve (linear curve corr.  $\geq 0.995$ , quadratic  $\geq 0.99$  with six points). The curve is then verified using a second source standard (**75-125% criteria**).

- b. The calibration curve must be verified every day through the analysis of a mid-level standard at the beginning and end of the sequence and after every 10 field samples. The percent difference back to the curve must not exceed  $\pm 20$  percent. If this criteria is not met, corrective action must be taken before sample analyses continues. Usually this involves recalibration or checking the gastight syringes.

c. Calculations:

$$\text{Calibration Factor (CF)} = \frac{\text{Response}}{\text{Dec Equiv} \times 1000}$$

Decimal equivalents are taken from the sample quant reports and entered into an Excel spreadsheet to calculate final concentration in ug/L.

2. Retention Time (RT) Windows - RT criteria set forth in SW-846 method 8000C are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration with midpoint standard. If the established retention time window is less than  $\pm 0.03$  minutes, the window defaults to  $\pm 0.03$  minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.
1. Quality control for this method can be referenced in SW-846 Method 8000C.
  - a. A method blank is required before analyzing samples. The contamination level should not exceed the CRDL.
  - b. An MS/MSD pair are required every 20 samples per matrix. Limits 75-125%.
  - c. A Laboratory Control Sample (LCS) is required every 20 samples. Limits 75-125%.
  - d. MDLs are either performed annually or by analyzing an MDL check according to SOP-414.

- C. Sample analysis includes the following steps: 15 ml of sample are transferred to 20ml headspace vials capped and loaded onto the autosampler along with a method blank with 15 ml of D.I. water.
1. A mid-level standard must be run at the beginning and end of the sequence and after every 10 field sample and cannot exceed  $\pm 20$  percent difference from the initial calibration. A mid-level standard must also be analyzed at the end of the analysis sequence.
  2. The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples.
- D. Following sample analysis, the data is reduced using the TARGET data system. The following must be checked to see if the samples will require re-analyses or dilution.
1. The analyte concentration must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.
- E. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with these methods. See SOP-413 for guidance.

## VII. HEALTH AND SAFETY

- A. Care should be used in handling all samples.
1. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
  2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
  3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

## VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION

A. Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## IX. REFERENCES

- A. Newell, Bryan, RSKSOP-175, Rev.0, August 1994.
- B. Newell, Bryan, RSKSOP-147, Rev.0, January 1993.
- C. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8000C.

**DEFINITIONS**

°C - degrees centigrade  
CF - calibration factor  
CRDL - contract required detection limit  
%D - percent difference  
FID - flame ionization detector  
GC - gas chromatograph  
LCS - laboratory control sample  
MDL - method detection limit  
μL - microliter  
μm - micrometer  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
%RSD - percent relative standard deviation  
RT - retention time  
SOP - standard operating procedure

Refer to SOP-431 for further definitions.

GC/MS SEMI-VOLATILE  
BNA-AQUEOUS MATRIX  
EXTRACTION USING  
SW-846 METHOD 3510C  
FOR 8270C/625 ANALYSIS

SOP NUMBER:

SOP-300

REVISION NUMBER:

17

APPROVED BY:

  
SECTION MANAGER

  
QUALITY ASSURANCE OFFICER

EFFECTIVE DATE:

09/23/08

DATE OF LAST REVIEW:

09/23/08

**GC/MS BNA - AQUEOUS MATRIX EXTRACTION  
USING SW846 METHOD 3510C/8270C, 625****I. SCOPE AND APPLICATION/SUMMARY**

1. This SOP describes the extraction of BNAs from water by separatory funnel extraction using SW846 Method 3510C and 625. Samples are extracted with methylene chloride and concentrated to an appropriate final volume.

**II. INTERFERENCES**

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**III. APPARATUS AND MATERIALS**

- Separatory Funnel - 2-Liter with Teflon stopcock
- Beaker - 400 mL
- Drying /Chromatographic column - 20 mm I.D. x 300 mm or funnel
- Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- Metal rack - capable of holding six glass evaporation tubes
- Turbo-Vap Evaporator - heated and capable of temperature control ( $\pm 5^{\circ}\text{C}$ ); the bath should be vented into a hood.
- Vials - 2 mL glass amber, with Teflon-lined screw cap and 40 mL with Teflon lid.
- pH indicator paper - close range (1.0 - 2.0) and (10.0 - 12.0); wide range (1.0 - 12.0)
- Syringe - 1 mL, 500 mL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Ring stand - 3 prong
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator

**IV. REAGENTS**

- Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.
- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
- Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in plastic bulk containers from Fisher # S415-10S or equivalent) placed in Pyrex tray and heated at 400°C for a minimum of 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.
- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).
- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H<sub>2</sub>SO<sub>4</sub> (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.
- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent).
- The GC/MS operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

**BNA Surrogate** - The base neutral and acid surrogates are normally mixed together in one solution. This solution is purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for surrogate amount of 100:200 ug/mL per sample. (**For low level PAHs use 1.0ml of a 1.0µg/mL BN Surrogate spiking solution.**)

**BNA Spiking Solution** - The base neutral and acid spiking solutions are normally mixed together in one solution ( **This spiking solution contains all the compounds that are normally calibrated by GC/MS** ). This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for LCS amount of 100ppm per sample. There are two separate spiking solutions available – one solution has a more complete list of BNA compounds than the other which is called the short or matrix spike list. The long list should be used on all extractions unless your supervisor has approved the short list. The short list may be used for any ‘phenol only’ extractions. (**For low level PAHs use 1.0 ml of a 1.0ppm of the LLPAH spiking solution.**)

**BNA TCLP Spike** – 0.5 mL is added per 100-mL volume. Each matrix type must have its own TCLP spike. TCLP spike should be added after the TCLP has been filtered but prior to refrigeration. From the volume provided by Wet Chemistry, remove a 100-mL aliquot into a suitable container with a teflon lid, and spike as indicated above.

## V. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
  - Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
  - Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
  - Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
2. Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

**SLUDGE** - use only 100 mL and dilute to 1000 mL with reagent water.

**TCLP EXTRACT** - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (which has already been spiked as explained in the BNA TCLP Spike section above) should be set up at the same time. Dilute to 1000 mL with reagent water.

**BAD MATRIX** – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. **SPLP extract- use 1 liter.**

**NPDES client** - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1 mL is added to the LCS and the matrix spike.

**ACID EXTRACT WITH BAD MATRIX** - a cleanup step is added. Samples are taken to a high pH, extracted with 60 mLs methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

3. **LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs)** – Samples require a BNA extraction. Use the Surrogate and BN spiking solutions specified. Low level PAHs are normally concentrated on the Turbo-Vap using Round-Bottom TV tubes to a final volume of 0.5 mL
4. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.
5. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume-Final Volume, and the Lab prebatch code. The lab batch code is defined as MMDDYYB# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.
6. Using the 1000-mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000 mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.
7. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

Generally 0.5 mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. **For low level PAHs use 1.0ml of a 1.0ppm LLPAH Surrogate spiking solution.** Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

For the sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe designated for BNA spike, to add 0.5 mL of BNA spiking solution. **For low level PAHs use 1.0 mL of the 1.0ppm LLPAHs spiking solution.** Someone must verify that the spike has been added by placing a check mark on each label as it is added. **For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.**

Enter the ID# of the surrogate/spike used and the initials of the person that verified their addition to the sample in the BNA logbook.

8. **ACID EXTRACTION:** Adjust the pH to between 1.0 and 2.0, using 2 mL of 1:1 H<sub>2</sub>SO<sub>4</sub>. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H<sub>2</sub>SO<sub>4</sub> solution in small increments, as required to attain the proper pH.
9. Add 40 mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.
10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.

**NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.**

11. Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.
12. Following Steps 9 and 10, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.
13. **BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater**, using 10N NaOH. Start by adding 5.0 mLs to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. **BNA extraction is necessary when doing low level PAHs.**

**NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.**

14. **FOR 8270 extraction:** Extract one more time with 40 mL of methylene chloride following Steps 9 and 10. Combine BN and Acid extracts in a same 400ml beaker, unless

the BN extract has large amount of emulsion; then it will be necessary to use a separate 400 mL beaker. Concentrate BN and acid extracts for one final extract.

**NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.**

**For 625 extraction:** extract 3 more times with 40 mL methylene chloride following steps 9 and 10. Combine BN extracts in separate 400 mL beaker. Concentrate BN and acid extracts separately for one final extract.

15. In the log book marked BNA extractions, enter the Client name, the Lab #, the date extracted, the initial volume, and 1.0 mL for the final volume and anything unusual that may have occurred with this sample. The final volume for low level PAHs is 0.5 mL.
16. Prepare to dry the sample by either of the following methods:
  - 16A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR
  - 16B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.
17. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.
18. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of

Na<sub>2</sub>SO<sub>4</sub> by sprinkling the top layer with Na<sub>2</sub>SO<sub>4</sub> until it hardens, separates, and drops to the bottom.

## 19. TURBO-VAP CONCENTRATION

Low level PAH sample concentration is primarily done by Turbo-Vap using Round-Bottom TV tubes.

- Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.
- Record the numbers of the Turbo-Vap tube in the BNA logbook and remove the tube to a metal holder. To help prevent cross contamination, place a piece of aluminum foil over the Turbo-Vap tube and punch a small hole in the top so that the nitrogen can be accessed.
- Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.
- Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. **THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!!** A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.25 mL of methylene chloride into a 0.50 mL syringe and add this aliquot to the centrifuge tube. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2-mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Compare the volume to a 2mL dummy vial containing 0.5 mL of

solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

20. Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume in the BNA logbook and the Avery label to the nearest 10-mL.
21. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

#### **VI. DOCUMENTATION OF CAPABILITY (DOC)**

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

#### **VII. WASTE MANAGEMENT AND POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### **VIII. METHOD PERFORMANCE**

Refer to SOP-201 for method performance.

#### **IX. HEALTH AND SAFETY**

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

#### **REFERENCES**

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 625.

#### **DEFINITIONS**

BNA- base/neutral acid  
°C - degrees centigrade

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COC - chain of custody  
DL - detection limit  
g - grams  
KD - kuderna danish  
LCS - laboratory control sample  
 $\mu\text{g/L}$  - micrograms per liter  
 $\mu\text{L}$  - microliter  
 $\mu\text{g/ml}$  - micrograms per milliliter  
ml - milliliter  
mm - millimeter  
MS - matrix spike  
MSD - matrix spike duplicate  
PAH- polynuclear aromatic hydrocarbon  
RL - reporting limit  
SOP - standard operating procedure  
v/v - volume to volume

Refer to SOP-431 for further definitions



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**EMPIRICAL LABORATORIES, LLC  
STANDARD OPERATING PROCEDURE**

**ORGANICS: SOP 338**

**REVISION #: 06**

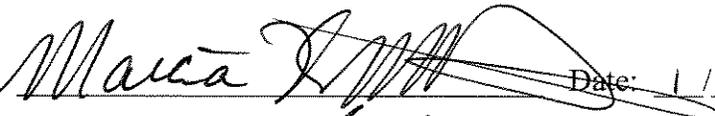
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**FLPRO  
METHOD FOR DETERMINATION OF PETROLEUM RANGE ORGANICS**

**APPROVALS:**

Lab Director:  Date: 1/21/10

Data Quality Manager:  Date: 1/21/10

Section Supervisor:  Date: 1/22/10

## **Changes Summary**

Revision Date: 1/22/10

- 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.

## Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
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11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
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15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
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# PETROLEUM HYDROCARBONS

## 1. TEST METHOD

1.1. This SOP is based upon method FL PRO.

## 2. APPLICABLE MATRIX

2.1. This SOP is applicable to the determination of the concentration of Petroleum Hydrocarbons in ground water, sediments, and wastes in the alkane range of C-8 to C-40.

## 3. DETECTION LIMIT

3.1. The detection limit for method FL-PRO is 0.085mg/L in water and 5.6 mg/Kg in soil.

## 4. SCOPE AND APPLICATION

4.1. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

4.2. This method is recommended for use by experienced analysts or under the close supervision for such qualified personnel.

## 5. SUMMARY OF METHOD

5.1. Samples are extracted via proper extraction methods. A 1µL aliquot of the extract is injected into a GC system equipped with a flame ionization detector (FID). Quantification is based on the detector response in comparison to a series of alkane standards.

## 6. DEFINITIONS

6.1. Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

6.2. Petroleum Hydrocarbons: All chromatographic peaks, both resolved and unresolved, eluting between the peak of n-octane (n-C8) and the peak end after n-tetracontane (n-C40). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from the FID response using baseline – baseline integration.

6.3. Petroleum Hydrocarbon Standard: A 17-component mix of all even-numbered alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

## 7. INTERFERENCES

7.1. All materials utilized during this analysis and the GC system must be demonstrated to be free from interference. Running frequent instrument blanks and methods blanks along with using pure, GC grade solvents will assist with the monitoring of interference's within the analytical system.

7.2. Any interference's co-extracted with the samples will vary considerably from source to source. Individual samples may require additional cleanup.

## 8. HEALTH AND SAFETY

- 8.1. Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab wide.

## 9. EQUIPMENT AND SUPPLIES

- 9.1 Separatory Funnel - 2-Liter with Teflon stopcock
- 9.2 Beakers- 250 ml
- 9.3 Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- 9.4 Metal or wood rack - capable of holding at a minimum six glass evaporation tubes
- 9.5 Turbo-Vap Evaporator - heated and capable of temperature control ( $\pm 5^{\circ}\text{C}$ ); the bath should be vented into a hood.
- 9.6 Silica Gel 60
- 9.7 Vials - 2 mL glass clear, with Teflon-lined screw cap
- 9.8 pH indicator paper - close range (0-6.0) and (7.0 - 14.0); wide range (1.0 - 12.0)
- 9.9 Syringe - 1000 $\mu\text{L}$
- 9.10 Graduated cylinder - Glass, Class A, 1000 mL
- 9.11 Pasteur pipette - length 9" and 5-3/4"
- 9.12 Pipette bulb
- 9.13 Aluminum foil - heavy duty
- 9.14 Nitrogen tank - equipped with pressure regulator
- 9.15 Ultrasonic Disrupter – capable of 300watts output, set on 10 Full power, pulse mode of 50%
- 9.16 A HP GC system, equipped with a flame ionization detector (FID), is used for analyzing extracts for all target analytes.
- 9.17 A Restek capillary column (P# RTX-5, 30m x 0.32mm x 0.25um) is used for analysis.
- 9.18 HP Chemstation Datasystem is used for data collection, detecting and storage.
- 9.19 Autosampler vials and caps appropriate to the sample tray are used for sample injection.
- 9.20 Microsyringes suitable for aliquoting 1.0  $\mu\text{L}$  to 1000  $\mu\text{L}$ s are used for standard preparation and sample dilution.
- 9.21 Class A volumetrics ranging from 1.0 ml to 250 mls are used for standard, spike and surrogate preparation.

## 10. STANDARDS AND REAGENTS

- 10.1. The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
  - 10.1.1. ASTM Type II Water.
  - 10.1.2. Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH into a 1L volumetric and cover with less than 1L reagent water. Use extreme caution when performing this step. Swirl the beaker until all pellets are dissolved (a stir plate can be used to mix the solution). This mixture gets very hot. Let stand until cool. Bring the solution up to the 1L mark with reagent water. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
  - 10.1.3. Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh placed in a Pyrex pan and heated at 400 $^{\circ}\text{C}$  overnight (minimum 4hrs), removed and cooled . Once cooled place in a labelled glass amber jar.
  - 10.1.4. Silica Gel 60 - Granular, anhydrous, trace pure 70-230 mesh. Weigh 60g in a 250 mL glass amber jar and add 1ml DI water to deactivate. Stored at room temperature.
  - 10.1.5. Glass Wool – Pre-rinse all glass wool used during the extraction process with Methylene Chloride.
  - 10.1.6. Sulfuric Acid Solution - (1:1), slowly add 500 mL of Sulfuric Acid to 500 mL of reagent water in a
  - 10.1.7. 1000 mL pyrex container. This mixture will get very warm. Allow to cool before use.
  - 10.1.8. Extraction Solvent - Methylene Chloride (Dichloromethane (Please read SOP-336 before using this solvent in our laboratory)- Omnisolv - suitable for spectrophotometry and gas chromatography (JT Baker) or equivalent.
  - 10.1.9. Carbon Disulfide– (Omnisolv - suitable for spectrophotometry, liquid chromatography and gas chromatography (JT Baker) or equivalent.
  - 10.1.10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

10.2. Stock Standards: Are non-Neat standards, received from vendors. These standards are used as intermediate standards to prepare working level standards. For unopened standards, if there is no expiration date assigned by the vendor, the expiration date must be assigned as 1 year from the date of receipt. For open stock standards, the expiration date is 6 months from the date the ampoule is opened or the vendor expiration date, whichever comes first. The following standards are used for the extraction and analysis of Petroleum Hydrocarbons:

<u>Vendor</u>	<u>Catalog #</u>	<u>Description/Conc</u>	<u>Used for Preparation of:</u>
Restek	31097	o-Terphenyl 10,000 ug/ml	Curve & Surrogate Soln.
Restek	31096	2-Fluorobophenyl 10,000 ug/ml	Curve & Surrogate Soln.
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc.=34,000ug/mL)	Spike Solution.
NSI	C-443-13	Florida TPH Mix 2000 ug/ml ea	Curve Solution
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc.=34,000ug/mL)	Alternate Source Verification Standard

*NOTE: The FL-PRO Petroleum Range Mix used for the preparation of the Spike Solution should always be a different Lot# from the Curve Standard.*

### 10.3 Working Standards

10.3.1 Are standards made from Neat or from stock standards, and are intended for analytical runs. The expiration date for these standards is 6 months from the date of preparation or the expiration of the parent stock, whichever date is first.

10.3.2 Follow analytical judgement when using standards. Evaluate standards on a daily basis versus past standards and instrument performance. A standard may evaporate or breakdown if proper storage processes are not used. Therefore, standards may have to be discarded before expiration dates.

## 11. SAMPLE 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. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

## 12. QUALITY CONTROL

12.1 An extraction batch must contain of no more than 20 client samples.

12.2 One BLK1, a BS1, BSD1, and a MS, MSD must be extracted in each batch.

12.3 Please follow guideline from Table 2 for meeting QC criteria.

12.4 All surrogates must pass the established laboratory criteria.

12.4.1 With samples requiring high level dilutions due to matrix interference or due to the abundance of target analytes, the surrogate will be diluted out and no recovery will be recorded. These samples can be reported.

12.4.2 For samples failing surrogate recovery high biased due to matrix interference, document the recoveries and notify the supervisor. In most cases, a Case Narrative should be filled out, the client should be notified, and the sample should be reported without a re-extraction. For samples failing the surrogate recovery (OTP) low biased, a re-extraction may need to be performed – check with supervisor. Any low recovery for surrogates

reported to client must be noted in case narrative and a CAR must be filled out. This is on a case by case basis and at the discretion of the department supervisor.

### 13. Calibration and Standardization

13.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization. See section 14.3 of this SOP.

### 14. Procedure

**14.1 AQUEOUS EXTRACTION:** All waters have a seven-day holding time. Determine the samples necessary to extract from the following sources. Note: never extract samples of unknown origin without discussion with supervisor):

- 14.1.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required
- 14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.
- 14.1.3 Periodically check LIMS throughout the day to determine what new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
- 14.1.4 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.
- 14.1.5 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid. Find out if any special dilutions are needed for the client. Routine procedures for difficult matrices are listed below.
- 14.1.6 BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made.
- 14.1.7 Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.
- 14.1.8 Set up enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples.
- 14.1.9 Place an Avery label on each separatory funnel containing the Lab #.
- 14.1.10 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. The lab batch code is generated by LIMS. The BLK1 and BS1 label should include all lab #s in this set of samples.
- 14.1.11 Mark the amber glass container of each sample at the water meniscus with “white out” or with a sharpie for later determination of sample volume. Determine the initial pH of sample and record on extraction sheet. If needed, adjust pH to between 1.0 and 2.0.
- 14.1.12 ACID pH Adjusting: Adjust the pH to between 1.0 and 2.0, using 1:1 H<sub>2</sub>SO<sub>4</sub>. Add the acid solution to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the pH using a 9” pipette with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H<sub>2</sub>SO<sub>4</sub> solution in small increments, as required to attain the proper pH. If sample is received without proper acid preservation, pH adjustment details must be recorded in LIMS.
- 14.1.13 Using the 1000-mL glass graduated cylinder measure 1000 mL of DI water and transfer it to a separatory funnel for each BLK1, BS1 & BSD1. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Rinse the sample bottle about 3-5 times with 10 mls aliquots of Methylene Chloride. Transfer this rinsate into the separatory funnel labeled with the sample ID.
- 14.1.14 Fill the sample bottle up to the mark with regular water. Now pour the water into a 1000 mL graduated cylinder. The volume measured is the initial volume to be documented for the sample in the LIMS. Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.

NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.

- 14.1.15 Add 50 mL of Methylene Chloride to each sample and to all the batch QC. Shake the sep funnel twice and vent into the hood. Repeat this venting process 3-4 more times and then manually shake the sep funnel for two minutes. Vent the sep funnel at the end of the two minutes. Some samples may require additional venting due to excess pressure buildup. Please use precaution with highly volatile and reactive samples. Place sep funnel, inverted, in shaker apparatus with stopcock open for 3 minutes.
- 14.1.16 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. Drain the bottom organic layer into a labeled 250 ml glass beaker first passing the extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride.
- 14.1.17 Follow Steps 14.1.15 and 14.1.16, two more times with 40 mLs of methylene chloride using the automatic shaker. Collect the extract from this step into the same beaker.
- 14.1.18 Transfer the extract to a pre-rinsed zymark tube by first passing through a funnel with glass wool and baked sodium sulfate all pre-rinsed with methylene chloride. After pouring the extract into the zymark tube, rinse the collection beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.
- 14.1.19 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
- 14.1.20 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- 14.1.21 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.
- 14.1.22 Sign the batch into the extraction laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.
- 14.1.23 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

**14.2 SOLID EXTRACTION** (may also follow extraction procedure outlined in SOP 343). All solids have a fourteen-day holding time counted. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):

- 14.2.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required
- 14.2.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.
- 14.2.3 Periodically check LIMS throughout the day to determine is new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
- 14.2.5 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.
- 14.2.6 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass jar. Routine procedures for difficult matrices are listed below:  
BAD MATRIX – for example a solid that is partially oil, see your supervisor to find out what dilution, if any should be made. Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set

- out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.
- 14.2.7 Get out enough 250mL beakers to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each extraction batch (up to a maximum of 20 samples). If sufficient sample is not available to perform a batch MS & MSD indicate such on the extraction sheet.
- 14.2.8 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. Label each 250mL beaker with the Lab ID.
- 14.2.9 Pre-weigh beakers and tare. Weigh 25g aliquot of the sample to the beaker and record weight to nearest 0.01g in extraction log. Add 25g dried Sodium Sulfate powder and stir the mixture well with a stainless steel spatula to a free-flowing sandy texture. If sample mixture forms large clumps, add more Sodium Sulfate to achieve proper texture (note in extraction log).
- 14.2.10 *It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*
- 14.2.10.1 *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- 14.2.10.2 *Two quarters should then be mixed to form halves.*
- 14.2.10.3 *The two halves should be mixed to form a homogenous matrix. This procedure should be repeated several times until the sample is adequately mixed.*
- NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.
- 14.2.11 Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.
- NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.
- 14.2.12 Add 60 mL of Methylene Chloride to each sample and to all the batch QC. Sonicate each sample for 3 minutes in Ultrasonic Disruptor (set on 10 Full power – pulse mode) at a pulse rate of 50%.
- 14.2.13 Decant the Methylene Chloride extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride, into a rinsed zymark tube.
- 14.2.14 Follow Steps 14.2.12 and 14.2.13, one more time with 60 mLs of methylene chloride. Collect the extract from this step into the appropriately labeled tube.
- 14.2.15 After pouring the extract into the zymark tube, rinse the beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.
- 14.2.16 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Note the turbovap pressure and temperature on the extraction logbook.
- 14.2.17 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
- 14.2.18 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- 14.2.19 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.

- 14.2.20 Sign the batch into the GC laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.
- 14.2.21 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

### 14.3 GCFID ANALYSIS

- 14.3.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.
- 14.3.2 Follow guidelines provided in the method for GC-FID conditions and sample volume to be injected for method FL Pro.
- 14.3.3 It is recommended that a solvent Blank be analyzed at the beginning of every sequence to ensure that the analytical instrument is free of contaminants.
- 14.3.4 All extracts within a batch are run on the Instrument after meeting calibration criteria as described in Table 2.
- 14.3.5 Qualitative and quantitative analysis is performed on samples.
  - 14.3.5.1 Qualitative Analysis for specific carbon ranges or fuel patterns, such as; mineral spirits, kerosene, JP-4 and heavy oils are performed per client request compared to specific standards. (See Table 3)
  - 14.3.5.2 Quantitative Analysis is performed using the following tools:
    - 14.3.5.2.1 FL PRO analysis is performed by running 6 calibration levels of a TPH mix from C-8 through C-40 (17 peaks). A response factor is calculated for each calibration standard (amount sum of 17 peaks/ std amount \* 17), then an Average Response factor is calculated for all 6 standards. This Average Response Factor is put in the method for uncalibrated peaks. Percent RSD must equal less than 20%.
    - 14.3.5.2.2 Surrogates o-Terphenyl and 2-fluorobiphenyl are added to each calibration standard at the same concentration. Initial calibration must pass acceptance criteria in Table 2.
    - 14.3.5.2.3 Analyte concentration must be within the calibration curve range. If the analyte concentration exceeds the calibration curve range, the extract must be diluted & rerun to bring the concentration within the calibration range. Use the calculation in section 15 to report the final results for the sample.
    - 14.3.5.2.4 Target analytes are calculated using the calibration curve and by incorporating any adjustments for initial or final volume and dilutions.

## 15. Data Analysis and Calculations

- 15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations throughout the laboratory.
- 15.2 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

The mean CF is calculated as follows:

- 15.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\text{Avg}CF} \times 100$$

- 15.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) \times 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV } CF - \text{Average } CF) \times 100}{\text{Average } CF}$$

- 15.5 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve. The concentration of a specific analyte is calculated as follows:

Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{[A_S] (V_t) (D)}{[\overline{CF}] (V_i) (D)}$$

where:

$A_S$  = Response for the analyte in the sample, units may be in area counts or peak height.

$V_t$  = Total volume of sample, mL.

$D$  = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made,  $D = 1$ .

$\overline{CF}$  = Mean calibration factor from initial calibration (area/ng)

$V_i$  = Volume of extract injected,  $\mu\text{L}$ .

$V_S$  = Volume of aqueous sample, mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is  $\mu\text{g/L}$ .

### Nonaqueous Samples:

$$\text{Concentration } (\mu\text{g/kg}) = [(A_s) (V_t) (D)] / [(CF) (V_i) (\overline{W_s})]$$

where:

$W_s =$  Weight of dry sample extracted, g.

$A_s$ ,  $V_t$ ,  $D$ ,  $CF$  and  $V_i$  have the same definition as for aqueous samples.

## **16. METHOD PERFORMANCE**

- 16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval.
- 16.2 See method FL-PRO for method performance.

## **17. Pollution Prevention**

- 17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **18. Data Assessment and Acceptance Criteria for Quality Control Measures**

- 18.1 Quality Control SOP QS05, "Data Deviations/Interpretations/Exceptions: Laboratory Non-Conformance/ Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results," provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

## **19. Contingencies for Handling out-of-control or unacceptable data**

- 19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

## **20. Waste Management and Pollution Prevention**

- 20.1 Please see Waste Disposal SOPs 210 and 405 for proper waste disposal.
- 20.2 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **21. References**

21.1 Method for Determination of Petroleum Range Organics (Method FL-PRO)

**22. Tables, Diagrams, Flowcharts and Validation Data**

Parameter	DL	LOD	LOQ/RL	LowCal
FL-PRO	0.085ug/L	0.17ug/L	0.34ug/L	0.17ug/L
FL-PRO	5.6ug/Kg	11.3ug/Kg	22.6ug/Kg	11.3ug/Kg

**Table 2 - Method Quality Control Requirements Summary**

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability									
Calibration Curve	<ul style="list-style-type: none"> <li>Prior to analyzing any samples</li> <li>A minimum of 5-points for linear fits</li> <li>A minimum of 6-points for quadratic fits</li> <li>Low standard at the RL/LOQ level</li> </ul>	<ul style="list-style-type: none"> <li>Linear correlation coefficient of at least 0.995</li> <li>Quadratic squared correlation coefficient of at least 0.99</li> <li>Average CF =&lt; 20% RSD</li> <li>Manual integrations on curve standards must have supervisory approval</li> <li>Must follow curve processing requirements from SOP QS08</li> </ul>	<ul style="list-style-type: none"> <li>Re-evaluate curve mix and makeup</li> <li>Re-run curve</li> <li>Check instrument for maintenance needs</li> <li>Re-prep the curve standards</li> </ul> <p>Samples cannot be analyzed until there is a passing calibration</p>									
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> <li>≤ 25% drift or difference for all analytes (≤ 20% for DoD samples)</li> </ul>	<ul style="list-style-type: none"> <li>Re-analyze an ICV from a different source</li> <li>Re-prep and re-analyze the ICV</li> <li>Re-calibrate and verify standard preps and sources</li> </ul>									
CCV	<ul style="list-style-type: none"> <li>At the beginning of every sequence</li> <li>For every 10-client samples and at the end of the sequence</li> <li>The concentration must be varied from low to mid range</li> </ul>	<ul style="list-style-type: none"> <li>≤ 25% drift or difference for all analytes (≤ 20% for DoD samples)</li> </ul>	<ul style="list-style-type: none"> <li>Evaluate the system for required maintenance</li> <li>Obtain passing CCV</li> <li>Reanalyze all samples injected since last passing CCV</li> <li>Q-qualify if reanalysis is not possible</li> </ul>									
MB	One per prep batch	<ul style="list-style-type: none"> <li>Must be less than ½ the LOQ.</li> </ul>	<ul style="list-style-type: none"> <li>Re-analysis to confirm the positive value</li> <li>Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers</li> <li>If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action</li> <li>Re-prep of samples associated with the MB</li> <li>NCR will be required for data reported</li> <li>Final Report data flagging will be required</li> </ul>									
Surrogates	Spike in every field or QC sample and standard	<table border="1"> <thead> <tr> <th>Surrogate</th> <th>Water</th> <th>Soil</th> </tr> </thead> <tbody> <tr> <td>Ortho-terphenyl</td> <td>30-140</td> <td>45-135</td> </tr> <tr> <td>2-Fluorobiphenyl</td> <td>50-150</td> <td>50-150</td> </tr> </tbody> </table>	Surrogate	Water	Soil	Ortho-terphenyl	30-140	45-135	2-Fluorobiphenyl	50-150	50-150	<ul style="list-style-type: none"> <li>Reanalyze to confirm recovery.</li> <li>Re-extract associated samples, if still failing</li> <li>Q-qualify if re-extraction is not possible</li> </ul>
Surrogate	Water	Soil										
Ortho-terphenyl	30-140	45-135										
2-Fluorobiphenyl	50-150	50-150										

**Table 2 - Method Quality Control Requirements Summary**

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
LCS	One per prep batch	Water 55-118% Soil 50-140%	<ul style="list-style-type: none"> <li>• Reanalyze to confirm recovery.</li> <li>• Re-extract associated samples, if still failing</li> <li>• Q-qualify if re-extraction is not possible</li> </ul>
LCSD	One per prep batch, when MS/MSD not included.	Water 55-118% Soil 50-140% RPD $\leq$ 30%	<ul style="list-style-type: none"> <li>• See LCS</li> </ul>
MS/MSD	One per prep batch, if sample volume available.	Water 55-118% Soil 50-140% RPD $\leq$ 30%	<ul style="list-style-type: none"> <li>• Reanalyze to confirm recovery.</li> <li>• Re-extract, if failure judged to be due to extraction.</li> <li>• J-qualify associated parent sample if re-extraction is not possible</li> </ul>
DOC Study	<ul style="list-style-type: none"> <li>• Initially per analyst prior to reporting data</li> <li>• Annually</li> <li>• Follow specific guidelines from section 16 for the preparation and analysis of DOC samples</li> </ul>	<ul style="list-style-type: none"> <li>• Must meet the criteria of the LCS for average recovery</li> <li>• Precision criteria is 20% standard deviation.</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or</li> <li>• Re-analysis</li> </ul>
LOD Verification	Every quarter	<ul style="list-style-type: none"> <li>• Parameter must be detected with response 3x the noise level</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Raise concentration</li> </ul>
LOQ Verification	Every quarter	<ul style="list-style-type: none"> <li>• Bias Requirement: Organics 50-150%</li> <li>• The LOQ value must be greater than the LOD value</li> </ul>	<ul style="list-style-type: none"> <li>• Re-prep and / or re-analysis</li> <li>• Raise concentration</li> </ul>
Retention Time Study	<ul style="list-style-type: none"> <li>• Prior to running samples</li> <li>• With major instrument changes (columns, etc.)</li> </ul>		

**Table-3**  
Qualitative Analysis Tool

SOP Title:

**BNA & Pesticide/PCBs & TPH NON-  
AQUEOUS MATRIX (MICROWAVE  
EXTRACTION) USING SW-846 METHOD  
3546**

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SOP NUMBER:

**SOP-343**

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REVISION NUMBER:

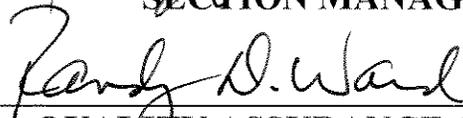
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APPROVED BY:

  
SECTION MANAGER

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QUALITY ASSURANCE OFFICER

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EFFECTIVE DATE:

**08/01/09**

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DATE OF LAST REVIEW:

**08/01/09**

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**BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX  
(Microwave Extraction)  
Using SW846 METHOD 3546**

**1. SCOPE AND APPLICATION**

- a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

**2. SUMMARY**

- a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

**3. INTERFERENCES**

- a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
- b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
- c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

**APPARATUS AND MATERIALS**

- d. Stainless Steel spatula
- e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
- f. Microwave extraction Teflon tubes, capacity approximately 75mL
- g. Suitable Teflon cap and screw-top lid
- h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
- i. Vial – 2mL clear with Teflon-lined screw cap
- j. Vial – 12mL clear with Teflon-lined screw cap
- k. Syringe – 1mL, 500uL
- l. Pasteur pipet – 9” length
- m. Pasteur pipet bulb
- n. Labels – Dymo
- o. Aluminum foil – heavy duty
- p. Nitrogen tank – equipped with pressure regulator
- q. TurboVap Concentrator with 200mL concentrator tubes
- r. Teflon funnels for pouring off
- s. Balance – capable of weighing to 0.1grams
- t. Aluminum pie pans for mixing samples
- u. Filter paper – 185mm

#### 4. REAGENTS

- a. Sodium Sulfate ( $\text{Na}_2\text{SO}_4$ ) – 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.

## 5. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
- b. Samples are preserved by cooling to 4°C.
- c. Holding time is 14 days from collection date to extraction.

## 6. PROCEDURE

- a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
  - i. Each day a backlog is generated in ELEMENT providing all relevant sample information, including samples numbers and respective analysis required.
  - ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
  - iii. Check the backlog throughout the day to re-evaluate priority if needed.
- b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
- c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
- d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
- e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container**

- f. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.
- g. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
- h. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.

NOTE: Surrogate and spike should be added just prior to extraction.

- i. Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH samples will need 1.0 mL of appropriate.

For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**

For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:

**Pesticide and PCB** - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

**Pesticide only** – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

**PCB only** - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na<sub>2</sub>SO<sub>4</sub>.

- j. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
- k. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.
- l. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
- m. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

For 1-15 samples:

Max power: 800W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

For 16-40 samples:

Max power: 1600W 100%

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

- n. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
- o. Transfer the extract to a pre-rinsed turbo vap tube by first passing through a funnel with P4 filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with Methylene Chloride and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.
- p. Now concentrate the extract to 1.0mL using the turbovap concentrator.
  - i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 50-55°C. The pressure target range should be about 20-25 psi.
  - ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
  - iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- q. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

## 7. DOCUMENTATION OF CAPABILITY (DOC)

- a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## 8. WASTE MANAGEMENT AND POLLUTION PREVENTION

- a. Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.
- b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

**9. METHOD PERFORMANCE**

- a. Refer to SOP-201 SOP-211 and SOP-219 for method performance.

**10. REFERENCES**

- a. EPA Methods SW-846, Method 3546

**11. DEFINITIONS**

- a. Refer to SOP-431 for definitions.

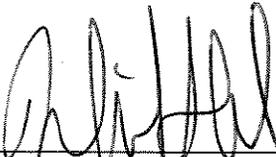
**12. HEALTH AND SAFETY**

- a. Wear appropriate personal protection equipment when working with chemicals or samples.
- b. Use the lab hoods when working with solvents.
- c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

**LABORATORY SAMPLE RECEIVING,**  
**LOG IN AND STORAGE**  
**STANDARD OPERATING PROCEDURES**

**SOP NUMBER:** SOP-404

**REVISION NUMBER:** 13

**APPROVED BY:**   
SECTION MANAGER

  
TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 06/29/09

**DATE OF LAST REVIEW :** 06/29/09

## LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

### SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the “Sample Receiving” section of this document.

### **I. Sample Receiving**

A. Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our

coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment II]* is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.
- B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."
1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.
  2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. **THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.**
- C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.
- D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign then list the date and time received on the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined for

leakage, breakage etc., the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.
  2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.
  3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.
- E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.
1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.
  2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

**The only exceptions to the 6°C rule are:**

- a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO<sub>3</sub> to a pH of  $\leq 2$ . *Keep in mind that non-aqueous sample for Metals must be cooled.*
  - b. Samples for Fluoride, Chloride and Bromide.
  - c. Waste/Product samples for all parameters.
  - d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.
  - e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received On Ice" (ROI).
  - f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.
- F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:
1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.
  - a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.
  - b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.
3. Samples requiring rush turnaround.
  - a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.
  - b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.
- G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.
  1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.
  2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.
- H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.
  2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.
- I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

## II. Sample Log In

- A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.
- B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.
1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)
  2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.
  3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).
- C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. ***When samples are unpacked they must be placed in cold storage within two hours even if they have not been through the entire log in procedure.*** All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)
2. The sample descriptions on the bottle should match those on the chain of custody. (See section IH)
3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO<sub>3</sub> used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.
  - a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.
  - b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather than a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

- c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH, Electrometric.]
4. The following guidelines must be followed to check pH preservation:
- a. Water samples for Cyanide analyses must be preserved to a pH of  $>12$  with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to  $\geq 12.0$  unless project/client specific requirements are to contact the client first.
  - b. Water samples for Metals analyses must be preserved to a pH of  $\leq 2.0$  with HNO<sub>3</sub> upon collection. If the pH of these samples is between 2.0-3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to  $\leq 2.0$ . unless project/client specific requirements are to contact the client first.
  - c. Samples requiring analyses which are preserved with H<sub>2</sub>SO<sub>4</sub> (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted  $\leq 2.0$  unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH  $>9$ .
  - d. If a sample is not properly preserved, log-in personnel must either do the following:
    - To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.
    - For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.
    - All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be CAR generated and the client notified to see if the lab is to proceed with analysis.

- e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).
  - f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.
5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.
  6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (0.2g) when chlorine is known to be, or suspected to be present.
  7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.
  8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.
  9. Be aware of holding time requirements. (See section 1D)

- D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VIII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:
1. Client and Log #s
  2. Date/Time Unpacked
  3. Logged In/Numbered By (Initials)
  4. 2nd Checked By (Initials)
  5. Date/Time Placed in Cold Storage
  6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
  7. Disposed of By/Date
  8. Method of Disposal
- E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

### III. Sample Storage

- A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***
1. The Hobart refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***
  2. Walk In Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.
- B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:
1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.
  2. All soils taken from foreign sources, US Territories and Hawaii.

**NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.**

- C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:
1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)
  2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.
- D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year (**digital thermometers quarterly**) and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

- E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the *Sample Custody Form [Attachment IX]* which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]
- F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.
- G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.
- H. A temperature maintenance record book is kept for each refrigerator.
- I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

#### **IV. Laboratory Information Management System (LIMS)**

- A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. ***Client:***

Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. ***Projects:***

Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

- a. Internal -- Empirical Laboratories projects;
- b. External -- direct laboratory clients.

3. **Comments:**

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. **Received By:**

Enter the name of the person who received the samples.

5. **Logged In By:**

Enter the name of the person who logged in the samples.

6. **Received:**

Enter the date and time received separated by a space and using military time.  
Example: 08/02/2008 08:30

7. **Project/Package Date Due:**

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. **Shipping Containers:**

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. **COC Number:**

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By:***

Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

*After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.*

11. ***Sample Name:***

- a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.
- b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.
- c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # \_\_\_\_". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:00 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, a CAR must be generated. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with

regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***pH (Container Preservative):***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

17. ***Comments:***

Enter any information that is applicable at the sample level.

18. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

19. ***Work Analyses:***

Select all parameters requested for the sample from this list.

- a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

- b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.
- c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

20. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

21. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

22. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

- a. A 2-digit numeral of the year. Example (0811248-06)
- b. A 2-digit numeral of the month. Example (0811248-06)
- c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)
- d. A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

23. ***Add/Edit/Copy:***

Use these selections to add more samples to the work order, or to change existing information prior to label printing.

*Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.*

- B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

#### V. Daily Follow Up for Sample Receiving/Log In

- A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.
- B. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).
- C. Complete any required CARs.
- D. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):
1. The original chains of custody and yellow original or copy of each. The CRF will accompany the COC for the project.
  2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.
  3. All CARs.
  4. LIMS sample receiving logs.
  5. Copies of any RUSH sheets which have been distributed
  6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.
- E. All the above information from the day will be reviewed as soon as possible.
1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.
  2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a *Sample Log Change Form [Attachment XIII]* through email distribution. A *Sample Log Change Form* by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.
- F. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:
1. Copies of the LIMS receiving reports to necessary laboratory personnel.
  2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.
  3. Finalized/approved CARs must be sent to the:
    - a. Organic Manager
    - b. Inorganic Manager
    - c. Laboratory Manager
    - d. Laboratory Director {optional}
    - e. Quality Assurance Officer
    - f. Administrative Assistant
    - g. Client {optional}
  4. Copies of any project/sample specific information to the Section Manager and analysts.
- G. Information will be filed as follows:
1. Chains of custody:

- a. Original (white copy) is returned to the customer with the final report along with the CRF.
  - b. Pink copies should be retained by the sampler.
2. CARs
- a. CARs can be found at V:\LAB\log-in\login (year)\logcar (year).
3. Sample Change Forms and RUSH Sheets
- a. Sample Change Forms are distributed by email.
  - b. RUSH Sheets are found at V:\LAB\login\Rushsheets
4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

## VI. Miscellaneous

- A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.
- B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.
- C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.
- D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as *Attachment XIV*.
- E. A *Telephone Conversation Log [Attachment XV]* may be required to document information and may be attached to or used as a CAR.

- F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

<b>Log Book ID</b>	<b>Log Book Description</b>
LI014	Trip Blank Prep Log Book
LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI013	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

**Attachments to SOP 404**

II	Chain of Custody Record
III	Corrective Action Report for Sample Receiving/Log In
IV	Cooler Receipt Form
V	List of Short Holding Time (Immediate-72 hrs.) Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the US.
IX	Laboratory Sample Custody Form for Walk In Refrigerator
X	Container Codes for the LIMS
XI	Routine NPDES Clients
XII	RUSH Sheet
XIII	Sample Log Change Form (Green Sheet)
XIV	Flow Chart, Laboratory Sample Tracking System

*[Attachments I and VI were removed during the editing process and not added to the SOP.]*



**EMPIRICAL LABORATORIES**

**CORRECTIVE ACTION REPORT FOR SAMPLE RECEIVING/LOG-IN**

**Date Completed:**

**Form Completed By:**

**Date Samples Received:**

**Parameter(s):**

**Client/Job #:**

**Samples:**

---

**Problem(s):**

**Action Taken:**

**Action Taken to Prevent Reoccurrence of this Problem:**

**Approval of Section Leader:**

---

**Distributed to:**



## Short Holding Time Parameters

(Immediate-72 hours)

Parameter	Holding Time
pH	Immediate <sup>a</sup>
Sulfite	Immediate <sup>a</sup>
Temperature	Immediate <sup>a</sup>
Residual Chlorine	Immediate <sup>a</sup>
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) <i>Drinking Water only</i>	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours <sup>b</sup>

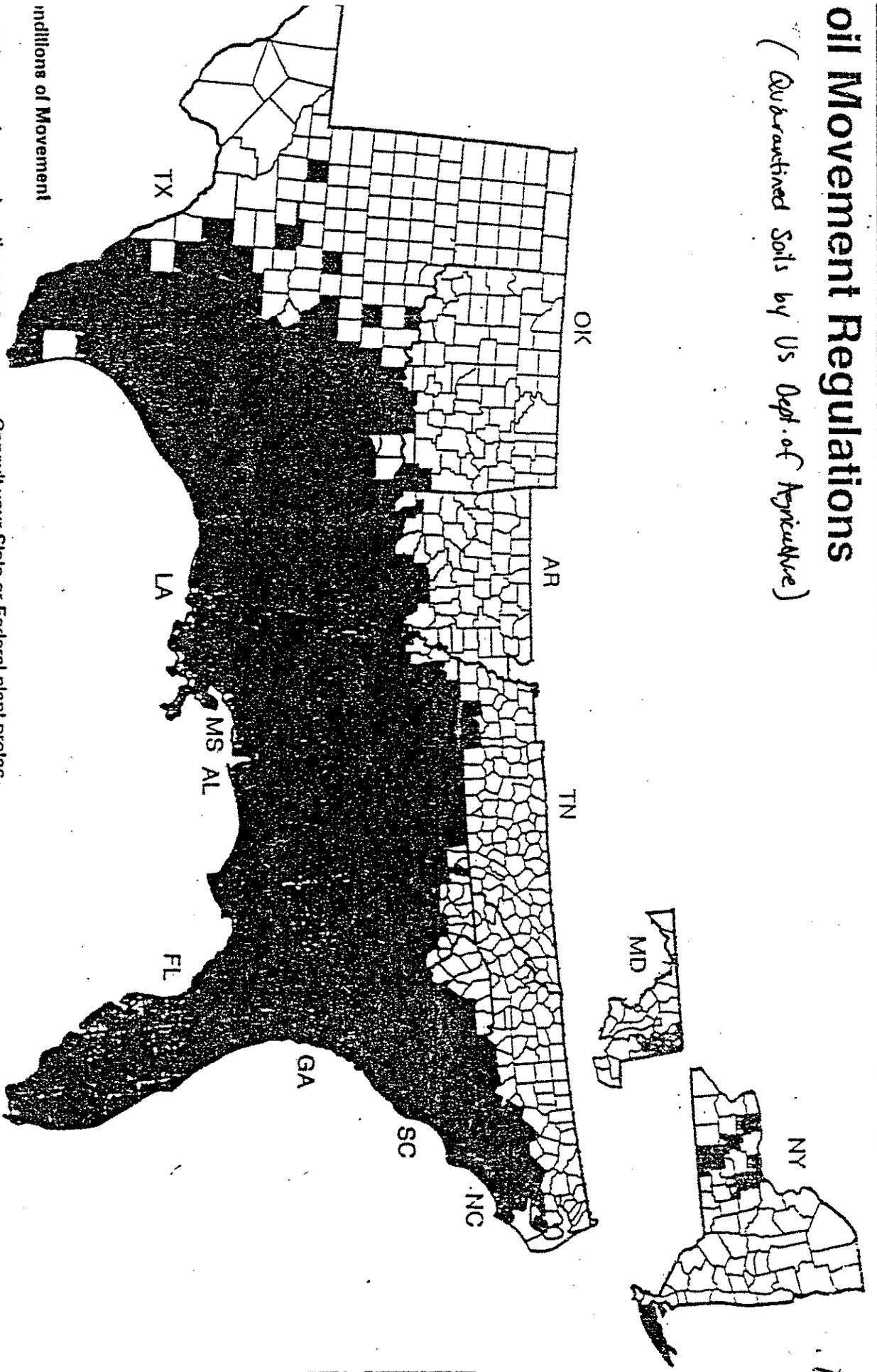
<sup>a</sup> Immediate generally means within 15 minutes of sample collection.

<sup>b</sup> This is an internal holding time. The method does not specify a holding time.



# Oil Movement Regulations

(Disadvantaged Soils by US Dept. of Agriculture)



ATTACHMENT VIII

## Conditions of Movement

Restrictions are imposed on the movement of regulated articles from a regulated area as follows:  
 1. From red areas into or through white areas.  
 2. From red areas into or through white areas. Movement within red areas may be regulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

 Regulated Area



<b>Preservatives</b>		<b>Types of Container</b>	
<b>NI</b>	<i>HNO3</i>	<b>A</b>	<i>1 LITER - PLASTIC</i>
<b>NF</b>	<i>HNO3 (Filtered)</i>	<b>B</b>	<i>500 mL - PLASTIC</i>
<b>SU</b>	<i>H2SO4</i>	<b>C</b>	<i>250 mL - PLASTIC</i>
<b>SH</b>	<i>NaOH</i>	<b>D</b>	<i>120 mL - PLASTIC</i>
<b>ZN</b>	<i>ZnAC / NaOH</i>	<b>EN</b>	<i>ENCORE PAK</i>
<b>HY</b>	<i>HCl</i>	<b>F</b>	<i>1 LITER - GLASS CLEAR WIDE MOUTH</i>
		<b>G</b>	<i>1 LITER - GLASS CLEAR BOSTON ROUND</i>
		<b>H</b>	<i>1 LITER - GLASS AMBER</i>
		<b>I</b>	<i>250 ml. - AMBER</i>
		<b>J</b>	<i>VOA VIALS - (40 ml.)</i>
		<b>K</b>	<i>500 ml. - (16 oz)</i>
		<b>L</b>	<i>250 ml. - (8 oz)</i>
		<b>M</b>	<i>125 ml. - (4 oz)</i>
		<b>N</b>	<i>60 ml. - (2 oz)</i>
		<b>O</b>	<i>OTHER</i>
		<b>P</b>	<i>PLASTIC BAG -1 Gallon</i>

ROUTINE NPDES CLIENTS (Page 1 of 2)

ALCAN Ingot and Recycling  
Amoco Oil  
Armstrong (Pirelli)  
Atochem-Carrollton, KY  
Auburn Hosiery Mill  
Autostyle

Bando Manufacturing  
Bowers Ink  
Bowling Green Municipalities (City of)  
BP Oil  
Bremner, Inc.  
Brentwood, City of  
Brown Printing Central

Burgill Steel and Wire  
Clarksville Products

Dupont  
Eaton and Olsen  
Emhart Pop Rivets

Fleet Design  
Franklin, City of

Gatlinburg

Hennessey Co. (Coats)  
H.I.S. Laundry  
H.K. Bell (City of Hopkinsville Landfill Monthly Monitoring)  
Hoover

International Paper  
J. S. Technos

Ken Koat  
King Industries

Lannom Tannery  
Leonard Plating

ROUTINE NPDES CLIENTS (Page 2 of 2)

al Plate, Inc.  
Morflex, Inc.

Nashville Wire  
Norandal USA Inc.

Oak Ridge, City of

Plymouth Tube  
Prime Colorants

RMI

Shared Hospital Services  
Snap On Tools  
Springfield, City of  
Special Metals  
Steel Industries

Tennessee Dickel Distillery  
Tulahoma, City of

UCAR, Clarksville

Valmore Leather

Westvaco (Mayfield Creek Up/Down)  
Woodbury

Revised 9/10/96



SAMPLE LOG CHANGE FORM

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DATE:

TO:

SAMPLE #(S):

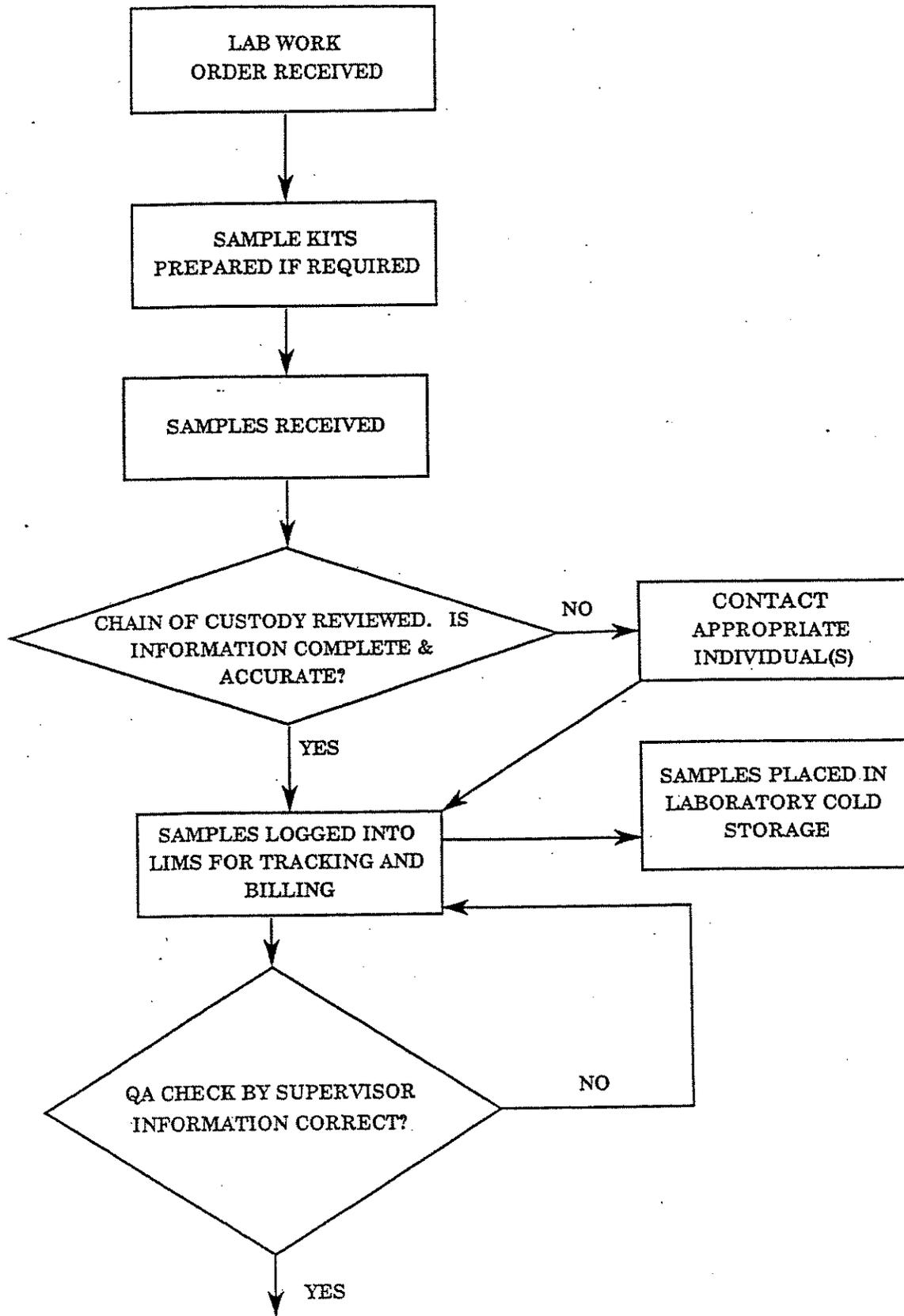
CLIENT:

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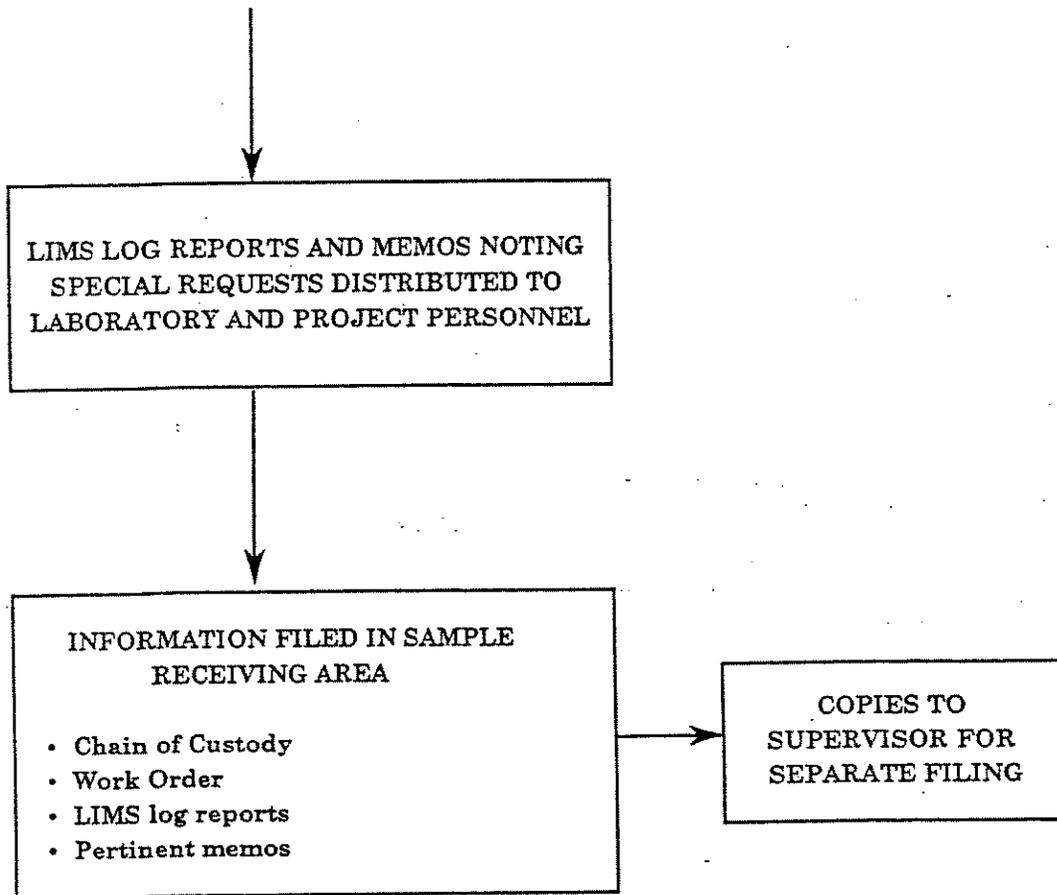
Changes:

LABORATORY SAMPLE TRACKING SYSTEM  
SAMPLE RECEIVING



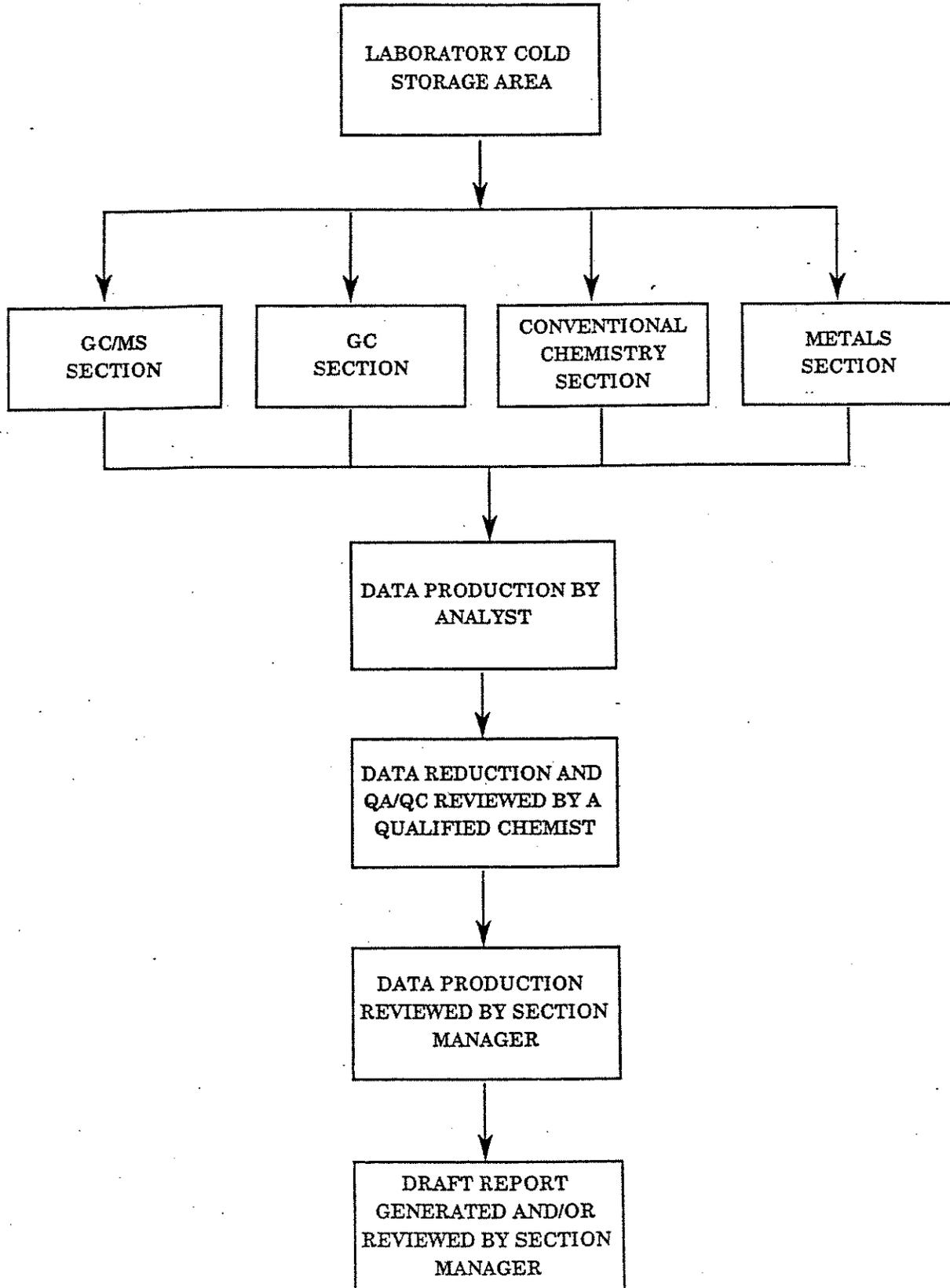
ATTACHMENT XIV (Continued)

LABORATORY SAMPLE TRACKING SYSTEM  
SAMPLE RECEIVING (continued)

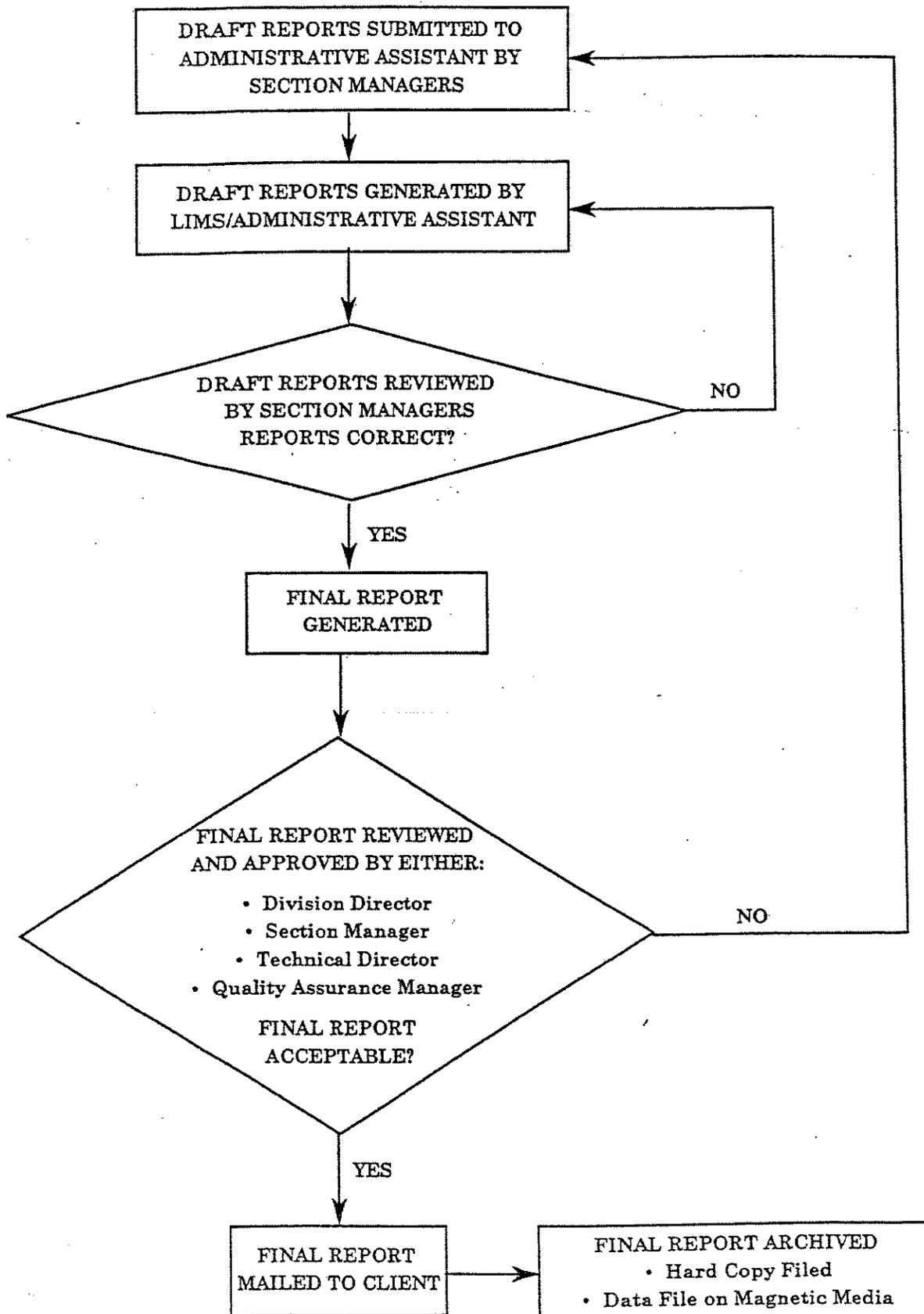


ATTACHMENT XIV (Continued)

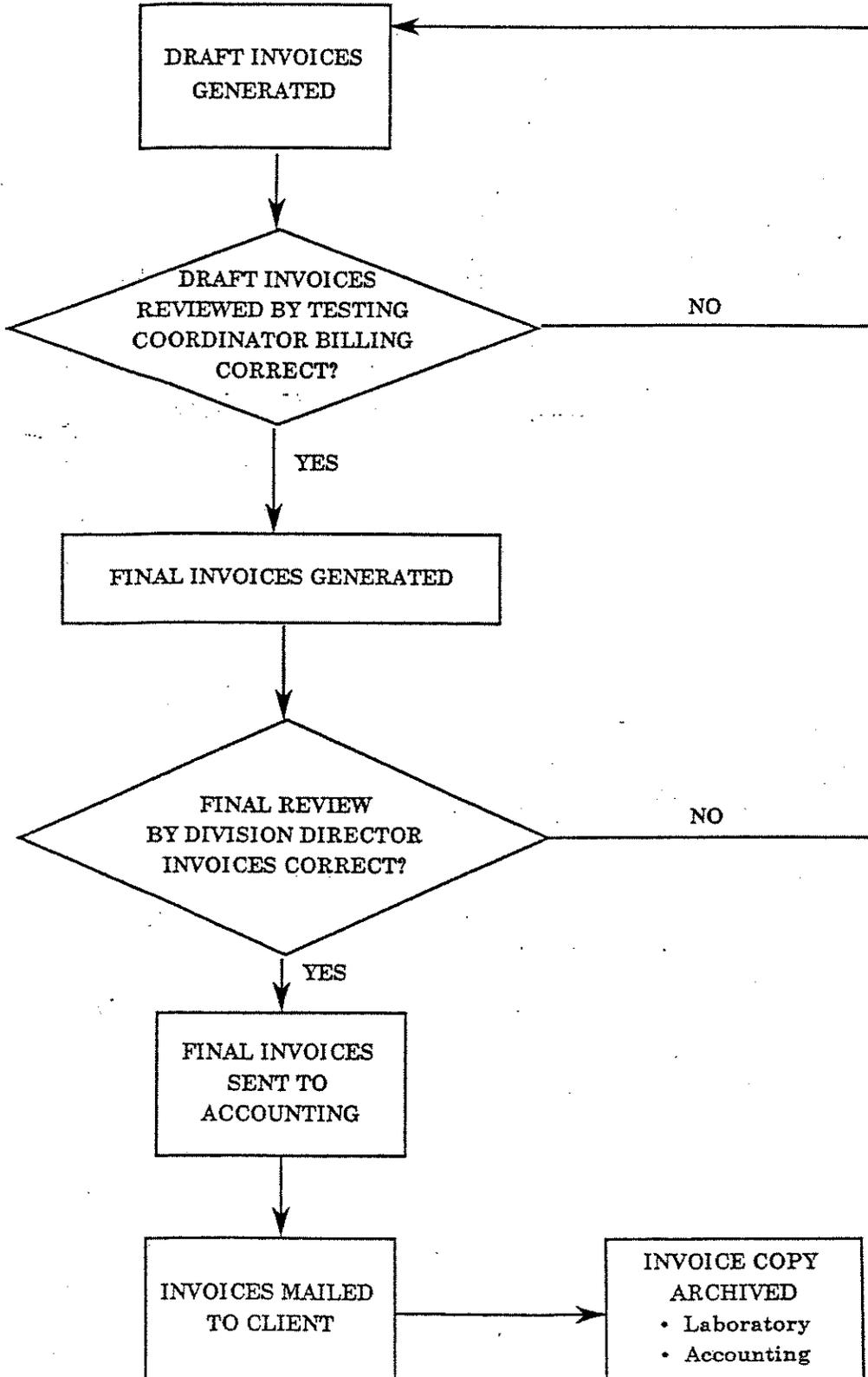
LABORATORY SAMPLE TRACKING SYSTEM  
DATA PRODUCTION AND REVIEW



LABORATORY SAMPLE TRACKING SYSTEM  
DRAFT AND FINAL REPORT



LABORATORY SAMPLE TRACKING SYSTEM  
INVOICING



# DISTRIBUTION/TRAINING LOG

**LABORATORY SAMPLE RECEIVING,  
LOGIN AND STORAGE  
STANDARD OPERATING PROCEDURES**

**SOP NUMBER:**

**SOP-404**

**REVISION NUMBER:**

**13**

**RECEIVED BY/DATE:**

W. Schwab

*W. Schwab* *WS*

F. Rivers

*FR* *FR*

~~R. Townsend~~

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

**TRAINED BY:**

*Landy D. Ward*

**EFFECTIVE DATE:**

**06/29/09**

**PLEASE COLLECT OLD SOPs AND RETURN WITH  
SIGNED FORM TO QAO**

**ANALYTICAL  
LABORATORY WASTE  
DISPOSAL**

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**SOP NUMBER:**

**SOP-405**

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**REVISION NUMBER:**

**5**

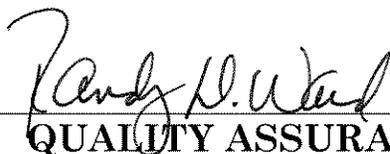
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**APPROVED BY:**



**LAB DIRECTOR**

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**QUALITY ASSURANCE  
OFFICER**

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**EFFECTIVE DATE:**

**06/23/09**

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**DATE OF LAST REVIEW:**

**06/23/09**

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## **Analytical Laboratory Waste Disposal Standard Operating Procedure**

### **I. SCOPE AND APPLICATION:**

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

#### **A. Wastes can be broken down into the following categories:**

1. Unused portions of actual samples received from outside clients.
  - a. Unused aliquots of completed water samples.
  - b. Unused aliquots of completed non-aqueous samples.
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

### **II. SUMMARY OF PROCEDURES:**

#### **A. There are four options for disposing of unused sample portions:**

1. Return completed samples and any generated waste from these samples to the client.
2. Throw the sample away after confirming that it is non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

**B. There are two options for disposing of laboratory generated waste:**

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

**For example: Solvent waste must be stored in the vented flammable cabinet.**

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

**III. EQUIPMENT/APPARATUS:**

**A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).**

**B. USDOT approved drums for storing and shipping hazardous waste.**

**C. Fume hood vented outside the building.**

**D. Flammable storage cabinet which is vented to the outside**

**IV. PROCEDURE**

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

**A. Disposal of completed aqueous samples:**

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.
  - a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.
  - b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.
  - c. **be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**
  - d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.
  - e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.
  - f. All samples disposed of in this manner must be documented in the bound disposal log.
2. If water samples are hazardous (known or suspected), one of the following steps must be taken.
  - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**
  - b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
  - c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

#### **B. Disposal of completed non-aqueous samples:**

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for two hours.

b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this manner must be documented in the bound disposal logbook with the following information:**

a. Client

b. Sample #s

- c. Date(s) treated
- d. Treatment method used

### C. Disposal of laboratory generated waste:

Generated waste is stored outside the building, inside the caged fence until a waste pick up occurs. This area must be maintained properly.

#### 1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

##### a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, (**>2% by volume**) and dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**
- Dilute acid waste (**≤2% by volume or less**) are neutralized using concentrated amounts of sodium hydroxide and poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated along with the amount of sodium hydroxide used to neutralize the acid waste, is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

##### b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed

below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards (**>2% by volume**) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ....etc...) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

**\*\*Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type and quantity of solvent is entered into the *'Organic Solvent Waste Logbook'* located on the shelf next to the drums.**

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located in the GC/HPLC Laboratory.

**PCB Box** – all samples/standards

**Pesticide Box** – all samples/standards

**Herbicide Box** – all samples/standards

**8330 Box** – all samples/standards

**Methylene Chloride Box**- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial. **Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

**Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

**Waste from the instrument** - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain (pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and disposed of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

**Standards - Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company.**

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.
- Hazardous or product samples are returned to the client.

#### **D. Consolidation of satellite waste for contractor disposal:**

In conjunction with the Safety Officer, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence (c. *Organic Extraction Laboratory Area*, \* **Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.
2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.
4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

**It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.**

# DISTRIBUTION/TRAINING LOG

## ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER:

SOP-405

REVISION NUMBER:

5

RECEIVED BY/DATE:

W. Schwab	<i>W. Schwab</i>	
R. Townsend	<i>Russell Townsend</i>	RET
F. Rivers	<i>F. Rivers</i>	FK
J. Holliman	<i>J. Holliman</i>	J.H.
B. DeVille	<i>Betty DeVille</i>	
A. Monteiro	<i>A. Monteiro</i>	
B. Richard	<i>B. Richard</i>	

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY:

*Randy H. Ward*

EFFECTIVE DATE:

06/23/09

**PLEASE COLLECT OLD SOPs AND RETURN WITH  
SIGNED FORM TO QAO**

**STANDARD OPERATING  
PROCEDURE (SOP) FOR  
LABORATORY SAMPLE  
STORAGE, SECURE AREAS  
AND SAMPLE CUSTODY**

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**SOP NUMBER:**

**SOP-410**

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**REVISION NUMBER:**

**7**

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**APPROVED BY:**

**SECTION MANAGER**

---

**TECHNICAL DIRECTOR**

---

**EFFECTIVE DATE:**

**06/23/09**

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**DATE OF LAST REVIEW:**

**06/23/09**

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**STANDARD OPERATING PROCEDURE (SOP) FOR  
LABORATORY SAMPLE STORAGE, SECURE AREAS  
AND SAMPLE CUSTODY**

Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Shipping/Sample Receiving (SR) area in back of the building. The laboratory is located close to Federal Express (FedEx) distribution station; therefore we pick up our coolers at the FedEx location daily and transport them directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client. The SR personnel must not leave any packages/cooler without authorized receipt from laboratory personnel. Samples must be accompanied by some type of chain of custody record. Sample receiving personnel sign, and list the date and time received on the chain of custody. The time received must reflect the actual time or validation date and time of receipt for the samples although they may be placed in cold storage and logged into the system at a later time. The method of delivery is listed on the cooler receipt form(CRF). The tracking # (if available) is attached to the chain of custody.

Once sample containers have been assigned a laboratory ID number, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A Cooler Receipt Form also must be completed to accompany the cohesive Chain of Custody. Samples should not leave the log-in area until this has been completed. Log-in is also responsible for maintaining a Sample Receiving Custody and Disposal Form for samples received. This form is to be filled out before the actual disposing of any

sample in house. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- Client and Log #s
- Date/Time Unpacked
- Logged In/Numbered By (Initials)
- 2<sup>nd</sup> Checked By (Initials)
- Date/Time Placed in Cold Storage
- Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- Disposed of By/Date
- Method of Disposal

Original samples are stored in following areas of the laboratory.

1. Hobart Refrigerator in the VOC lab: All water VOCs must be stored in this refrigerator.
2. Walk In Refrigerator: All waters for all analyses except must be stored in this refrigerator.
3. Soil Walk in Refrigerator for all soils.
4. Sample Archive Room: All samples that have parameters where holding times have already expired may be kept in this room. This is only utilized when the water walk in refrigerator is completely full of samples within holding times that have not expired.

All soils are treated as quarantined.

All samples must be stored in one of the four locations detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples which could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Testing Coordinator for other options.

Any person removing samples from the storage areas listed above, must sign them out on a laboratory custody sheet (attached). The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

Sample extracts and digestates are stored in the following areas:

1. All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.
2. Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.
3. Extracts from medium level VOC analyses are also stored in the Soil Walk – in or VOC sample freezer in the VOC Lab.
4. All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure. Samples will be held for a minimum of 30 days after the final report unless specified otherwise. Sample extracts and digestates are held for a minimum of 60 days after the final report unless project specific requirements state otherwise. See SOP No. 405 entitled Laboratory Waste Disposal SOP for guidance on disposal of samples.

The following personnel as of June 23rd, 2009 have access to all sample storage areas:

James Dalton	Herbie Johnson
Ashley Bester	Dahae Kim
Roger Burr	Dustin Lynch
Janice Shilling	Marcia McGinnity
Rick Davis	Badeen Mekael
Jessica Sales	Antonio Montiero
Betty DeVille	Kelienne Verdier
Amanda Fei	Gino Moore
Kendra Gentry	Lorraine Norohna
Jason Goodman	Melanie Sams
Sonya Gordon	Brian Richard
Gwen Hallquist	Franklin Rivers
Veronica Mullen	William Schwab
William Lancaster	Russell Townsend
Jade Holliman	Christy Thompson
John Hughes	Renee Vogel
Karu Huka	Randy Ward

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

For additional information see SOP No. 404 entitled Laboratory Sample Receiving, Log-In and Storage.

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>WC_ANIONS_300.0 (Regular) in Water (E300.0)</b>								
<b>Preservation:</b> Cool4_Cooled to 4°C								
<b>Container:</b> 0500P_NP(500mL Plastic			<b>Amount Required:</b> 5.0		<b>Hold Time:</b> 28 days			
Unpreserved)								
Bromide	0.0420	0.125 mg/L		20	80 - 120	20	90 - 110	
Chloride	0.170	0.500 mg/L		20	80 - 120	20	90 - 110	
Fluoride	0.0330	0.100 mg/L		20	80 - 120	20	90 - 110	
Sulfate as SO4	0.330	1.00 mg/L		20	80 - 120	20	90 - 110	

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>WC_ANIONS_300.0 (Short Hold) in Water (E300.0)</b>								
<b>Preservation:</b> Cool4_Cooled to 4°C								
<b>Container:</b> 0500P_NP(500mL Plastic			<b>Amount Required:</b> 5.0		<b>Hold Time:</b> 2 days			
Unpreserved)								
Nitrate as N	0.0330	0.100 mg/L		20	80 - 120	20	90 - 110	
Nitrite as N	0.0330	0.100 mg/L		20	80 - 120	20	90 - 110	

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>WC_TOT_PHOS_4500PB5E in Water (SM4500PB5E)</b>								
<b>Preservation:</b> H2SO4_Preserved with Sulfuric acid								
<b>Container:</b> 0500P_H2SO4(500mL Plastic			<b>Amount Required:</b> 50mL		<b>Hold Time:</b> 28 days			
w/Sulfuric Acid)								
Phosphorus, Total (as P)	0.0200	0.0600 mg/L		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>SGC_FLPRO_3546 in Solid (FLPRO)</b>								
<b>Preservation:</b> Cool4_Cooled to 4°C								
<b>Container:</b> 0004J_NP (4oz Jar Unpreserved)								
<b>Amount Required:</b> 15								
<b>Hold Time:</b> 14 days								
Petroleum Range Organics	11.0	33.0 mg/Kg			50 - 140	40	50 - 140	40
surr: 2-Fluorobiphenyl			50 - 150					
surr: o-Terphenyl			35 - 140					

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>WC_ALKALINITY_2320B in Water (SM2320B)</b>								
<b>Preservation:</b> Cool4_Cooled to 4°C								
<b>Container:</b> 0500P_NP(500mL Plastic			<b>Amount Required:</b> 25mL		<b>Hold Time:</b> 14 days			
Unpreserved)								
Alkalinity (as CaCO <sub>3</sub> )	1.00	1.00 mg/L		20	75 - 125	20	80 - 120	20
Alkalinity, Bicarbonate (as CaCO <sub>3</sub> )	1.00	1.00 mg/L		20	75 - 125	20	80 - 120	20
Alkalinity, Carbonate (as CaCO <sub>3</sub> )	1.00	1.00 mg/L		20	75 - 125	20	80 - 120	20
Alkalinity, Phenolphthalein (as CaCO <sub>3</sub> )	1.00	1.00 mg/L		20	75 - 125	20	80 - 120	20
Alkalinity, Total (as CaCO <sub>3</sub> )	1.00	1.00 mg/L		20	75 - 125	20	80 - 120	20

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike / LCS %R	Blank Spike / LCS RPD
<b>WC_ALKALINITY_2320B in Solid (SM2320B)</b>								
<b>Preservation:</b> Cool4_Cooled to 4°C								
<b>Container:</b> 0004J_NP (4oz Jar Unpreserved)			<b>Amount Required:</b> 10g		<b>Hold Time:</b> 14 days			
Alkalinity (as CaCO <sub>3</sub> )	10.0	10.0 mg/L		25	80 - 120	20	75 - 125	20
Alkalinity, Bicarbonate (as CaCO <sub>3</sub> )	10.0	10.0 mg/L		25	80 - 120	20	75 - 125	20
Alkalinity, Carbonate (as CaCO <sub>3</sub> )	10.0	10.0 mg/L		25	80 - 120	20	75 - 125	20
Alkalinity, Phenolphthalein (as CaCO <sub>3</sub> )	10.0	10.0 mg/L		25	80 - 120	20	75 - 125	20
Alkalinity, Total (as CaCO <sub>3</sub> )	10.0	10.0 mg/L		25	80 - 120	20	75 - 125	20

Method	Matrix	Analyte	Limits
SW8270D	Water	Acenaphthene	41-132
SW8270D	Water	Acenaphthylene	43-140
SW8270D	Water	Anthracene	50-139
SW8270D	Water	Benzo (a) anthracene	58-141
SW8270D	Water	Benzo (a) pyrene	31-142
SW8270D	Water	Benzo (b) fluoranthene	42-156
SW8270D	Water	Benzo (g,h,i) perylene	12-171
SW8270D	Water	Benzo (k) fluoranthene	49-165
SW8270D	Water	Chrysene	51-155
SW8270D	Water	Dibenz (a,h) anthracene	28-153
SW8270D	Water	Fluoranthene	47-158
SW8270D	Water	Fluorene	40-140
SW8270D	Water	Indeno (1,2,3-cd) pyrene	20-167
SW8270D	Water	1-Methylnaphthalene	35-131
SW8270D	Water	2-Methylnaphthalene	36-121
SW8270D	Water	Naphthalene	39-125
SW8270D	Water	Phenanthrene	46-144
SW8270D	Water	Pyrene	39-158
SW8270D	Solid	Benzo (a) pyrene	28-128
SW8270D	Solid	Benzo (b) fluoranthene	30-139
SW8270D	Solid	Benzo (g,h,i) perylene	21-149
SW8270D	Solid	Benzo (k) fluoranthene	42-129
SW8270D	Solid	Chrysene	39-134
SW8270D	Solid	Dibenz (a,h) anthracene	30-138
SW8270D	Solid	Fluoranthene	30-142
SW8270D	Solid	Fluorene	27-116
SW8270D	Solid	Indeno (1,2,3-cd) pyrene	17-164
SW8270D	Solid	1-Methylnaphthalene	30-111
SW8270D	Solid	Acenaphthene	28-110
SW8270D	Solid	Acenaphthylene	23-126
SW8270D	Solid	Anthracene	28-136
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## **MICROBIAL INSIGHTS**

## STANDARD OPERATING PROCEDURE

### STANDARD OPERATING PROCEDURE for Sample Receiving

SOP Number: MI SOP- SAMREC

Revision Number: 1.1 FINAL

Effective Date: 11/14/2008

MI Controlled Document

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MI Controlled Document

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure is to outline the steps involved in sample receipt and storage.

## 2.0 DEFINITIONS

**Aliquot:** A portion of a sample

**Holding Time:** The maximum time that a sample may be held prior to analysis

**LIMS:** Laboratory Information Management System

**Non-conformance:** Sample documentation that is received with incorrect, incomplete, or inadequate information.

**Preservation:** Refrigeration and/or reagents added prior to sample collection to maintain the chemical, physical and/or biological integrity of a sample.

**Short Holding Time:** Samples must be analyzed within 48 hours or less

**Temperature Blank:** A sample bottle of water that accompanies the samples in each cooler. This blank is used to monitor cooler temperature upon receipt of samples.

**Trip Blank:** A set of 40mL VOA vials filled with de-ionized water that travels with the samples to be analyzed for Volatile Organic Compounds (VOC). These samples are analyzed to determine if cross contamination occurred during sampling.

## 3.0 RESPONSIBILITIES

### 3.1 Sample Custodian

The responsibility of the Sample Custodian is to sign and date all appropriate receiving documents including Chain of custody (COCs) and shipping papers. Signing the COC indicates that the laboratory accepts the samples in their condition upon arrival. Conditions under which COCs will be signed or left unsigned are discussed in Section 4.0 of this SOP. The Sample Custodian is also responsible for notifying the client of all the discrepancies that occur during the sample receipt process. In each case, the client shall be consulted to resolve all discrepancies involving their samples. The Sample Custodian shall log samples in accurately and in a timely manner. The sample Custodian reports to the Director or Laboratory Supervisors.

### 3.2 Reporting Level Requirements and Responsibility

Microbial Insights' Director or Laboratory Manager(s) have the responsibility to alert the Sample Custodian, in writing or through the Laboratory Information Management System (LIMS), of any projects that require more than standard reporting requirements prior to delivery of those samples. Quality control reporting levels other than standard must be arranged with Microbial Insights Director or Laboratory Manager(s) prior to delivery of the samples. The

Director or Laboratory Manager(s) will then serve as the Project Manager for that project. If notification is not made prior to sample receipt, the level of reporting requested by the client cannot be assured.

### 3.3 General Documentation Requirements and Responsibility

The responsibility for quality control and documentation during sample receiving requires a diligent effort to ensure that all documentation is present and complete. The Sample Custodian is responsible for the sample receipt documentation and for client contact and documenting client responses in the event of non-conformance.

All records must be written in ink or printed electronically. When any changes are made, a single line should be drawn through the errors and the corrections to be made and initialed and dated. All notations written on COCs shall be dated and initialed.

## 4.0 SAMPLE RECEIPT

The following policy and procedures are in place to ensure that all samples and COC forms that are accepted at Microbial Insights are thoroughly inspected and all discrepancies are fully documented. The Sample Custodian and/or Laboratory Manager(s) shall contact the client in the event that there is any discrepancy involved in the condition or the documentation of samples, upon receipt, that may affect the sample's integrity or the analytical process.

### 4.1 Sample Receipt Record

A permanent record of sample receipt shall be maintained electronically in the LIMS. At a minimum, that record will include (1) Client Name; (2) Project Name; (3) Date and time of sample receipt; (4) Unique laboratory identification; (5) Name or initials of the person making the entries; (6) Requested analyses.

### 4.2 Sample Acceptance Policy

- Samples that are shipped to Microbial Insights must be accompanied by proper full and complete documentation. This documentation shall be marked on a COC and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type (if needed), sample matrix, specific parameters to be analyzed, and any other pertinent information to that sample set.
- Sample labels shall be supplied by Microbial Insights or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the COC form.
- Samples must be received within the specific holding times. Clients are requested to notify Microbial Insights Sample Custodian if unsure of the holding times.

- Samples must arrive at Microbial Insights with sufficient volume to conduct the requested analyses (All bottles should be filled completely, if possible).
- When problems with samples or documentation are found during the sample receiving process, the Sample Custodian will make every attempt to contact the client as soon as possible to make decisions concerning those discrepancies.
- In the event the client cannot be reached, a message will be left either on voice mail, a brief message with the receptionist to return the call, or by an email. Samples will be placed in a storage refrigerator and held until Microbial Insights gets a response from the client. (Exceptions will be made when samples are received that have short holding times and the samples are from a client with whom Microbial Insights has regular and frequent dealings. Also when the samples have short holding times and the samples are from a client with whom Microbial Insights has a signed contract, work order or purchase order).

#### 4.3 Non-Conformance Notification

The corrective action form should be filled out and the client should be notified in case of any of the following occurrences:

- Temperature discrepancy
- Hold time discrepancy
- Broken, leaked or missing samples
- Insufficient sample volume

Any analyses that are conducted on samples that do not meet these acceptance criteria will be noted within the client data folder, Microbial Insights LIMS, and noted on the front cover sheet of the client's final report.

#### 4.4 Sample Rejection Criteria

The following situations dictate when samples will be rejected:

- Coolers and samples arrive at Microbial Insights' facility with no client information
- Coolers do not arrive on time at Microbial Insights' facility due to error by the carrier (ie. Fedex, UPS or DHL) in which samples have strict holding times.
- Samples arrive with no COC form and there is no means to obtain one.
- Coolers that arrive with hazard labels on them for which Microbial Insights is not equipped or certified.

#### 4.5 Sample Receipt Procedure

- 
- 4.5.1 Prior to signing electronic shipping document from courier, ensure that the number of packages listed on the electronic document corresponds with the number of packages actually delivered.
  - 4.5.2 Remove any air bills from the outside of the coolers, place in the designated data folder assigned to that project set.
  - 4.5.3 Put on safety glasses and protective gloves before handling, opening, or unpacking packages and coolers that contain environmental samples.
  - 4.5.4 Retrieve the COC form from the inside of cooler and set aside or place in designated data folder.
  - 4.5.5 Place thermometer in cooler and record cooler temperature, note on designated form, initial and date entry. The temperature in the cooler should be  $4^{\circ}\text{C} \pm 2$  degrees. If the temperature is not within those parameters, note the discrepancies on the designated form and include it with the data folder. Also, bring to the attention of the Laboratory Managers the temperature for any further decisions regarding the validity of the samples.
  - 4.5.6 Inspect each sample and label while removing it from the cooler. Sample containers or filter packages should be intact. At a minimum, sample labels should be completed with the following information:
    - a. Sample Name/Number
    - b. Date and Time of Collection
    - c. Location
    - d. Analyses to be done
  - 4.5.7 If samples were received in grouped sets, keep the sets grouped together as they are unpacked. If samples were not received in sets, organize them into sets while unpacking.
  - 4.5.8 Match the sample identifications to the COC form. Note any discrepancies on the COC and with the data set folder.
  - 4.5.9 Make sure that field and trip blanks (if needed) are present and identified. Document all missing or potentially missing samples on the COC form and in the data set folder.
  - 4.5.10 Check the COC form to ensure that all samples are entered and the specific analysis is checked appropriately for each bottle, filter, or Bio-Trap.
  - 4.5.11 Ensure that all sample receipt documentation is complete, sign the COC form.

#### 4.6 Weekend Sample Receipt Procedure

All coolers shipped for weekend delivery are signed for by a Microbial Insights employee and stored in the storage appropriately until normal business hours Monday morning.

Water samples have to be stored in a refrigerator, all other samples need to be stored in a

freezer. Occasionally, laboratory technicians will work on weekends and will conduct an abbreviated sample receipt process, and the complete process will occur during normal business hours Monday morning.

## 5.0 SAMPLE STORAGE

Samples shall be stored according to the conditions specified by preservation protocols. The storage conditions shall be maintained, monitored, and documented. Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in segregated areas to prevent cross contamination.

### 5.1 Sample Storage Temperature

- 5.1.1 Samples which require thermal preservation shall be stored under refrigeration. It is the Laboratory Technician(s) responsibility to ensure that the refrigerator temperatures within the sample reception area are monitored and recorded on the Temperature Log Sheet. The Temperature Log Sheet is available within each laboratory.
- 5.1.2 If the temperature of the refrigerator/ freezer is outside of the acceptable range limits, the Laboratory Technician(s) must immediately notify the Laboratory Manager(s) and the Company Manager. Maintenance will be arranged through the Laboratory Manager or his or her representative. If it is apparent that the proper sample temperature cannot be maintained in the area needing maintenance, then every effort will be made to move samples to another refrigerator that is functioning within the temperature control limits.

## 6.0 SAFETY

Personnel safety is a priority at Microbial Insights. All employees are required to wear appropriate personal protective equipment in accordance with Microbial Insights Chemical Hygiene Plan. The Sample Custodian has been provided with safety glasses, protective gloves and a laboratory coat.

It is required that the Sample Custodian wear gloves and safety glasses while handling all coolers and samples. Coolers shall be opened in a well-ventilated area. If odors are detected upon opening, the cooler shall be closed and moved to the fume hood in the Commercial Laboratory before proceeding with the sample receipt procedures.

## 7.0 COOLER STORAGE AND DISPOSAL

Coolers shall be emptied of any melted ice and left to air dry. Coolers are then stored in a designated storage area. For any cooler that has aqueous residue from a sample container, proper disposal of the aqueous waste is followed then the cooler is rinsed, air dried and stored.

Any coolers which are damaged are then stored in separate storage section to be later discarded.

## **STANDARD OPERATING PROCEDURE**

### **Extraction of DNA from environmental samples (Matrix-Water, Soil, Biofilm, Bio-Sep Beads)**

**SOP Number:** MI SOP-DNA  
EXT

**Revision Number:** 1.0 FINAL

**Effective Date:** 1/05/06

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## ATTACHMENTS

Attachment 1: Laboratory Worksheet

## PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the activities involved in the preparation, handling, documentation, and extraction of nucleic acids of samples collected from a variety of matrices (water, soil, bio-traps, etc).

### 1.0 SCOPE AND APPLICATION

The extraction process involves both chemical and mechanical lysis of cells to release chromosomal and plasmid nucleic acids. After the extraction, Microbial Insights, Inc. (MI) uses these products in a variety of analyses.

### 2.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

Samples should be collected in field following Microbial Insights, Inc. (MI) standard operating procedure for sample collection. Samples must be maintained at 4°C and extracted within 24-48 hours of collection. DNA extractions will be held at -20°C for at least 1 year in case there is a need for additional analysis.

### 3.0 EQUIPMENT AND SUPPLIES

- 
- Micropipetters (Gilson P-10, P-100, P-1000) and appropriate sterile tips (ART). [See pipet calibration and maintenance in calibration log.]
  - Sartorius AG Analytical scale, Model BP310. (Sartorius) [See scale calibration and maintenance in calibration log]
  - Sterile spatulas and transfer paper.
  - Microcentrifuge Eppendorf model #5415D.
  - Vortex Mixer (MO BIO Adapter) .

### 4.0 REAGENTS



## 5.0 TESTING PROCEDURE

### 5.1 Sample Preparation:

#### 5.1.1 Cell Cultures

If the sample consists of bacterial colonies on plated media, use a flame sterilized inoculating loop to pick a colony from the plate. If the sample is a liquid culture, use a sterile 1ml syringe to remove 500µl of sample. Place the sample into the 2ml bead solution tube.

#### 5.1.2 Water samples

Filter the water through a 0.2 micron Supor filter. Filter the entire volume of water received until the filter becomes clogged. If less than 250mls of water has been filtered, a second filter should be used. Record the volume of water filtered, and discard the filtered water. Place the filter/filters into a sterile Petri dish and leave at 4°C until the sample is processed. For processing, use a sterile razor blade to cut the filter into sections and place into the 2ml bead solution tube.

#### 5.1.3 Soil, solid and Biofilm samples

Use a flame sterilized spatula to place 0.5 grams of the soil/solid/biofilm sample into the 2ml bead solution tube.

#### 5.1.4 Bio-Sep Bead samples

Use flame sterilized tweezers to place 20 Bio-Sep beads into the 2ml bead solution tube.

### 5.2 Extraction process

5.2.1. Label 2ml bead tubes with sample name and number. For every extraction set a blank tube must be labeled with the set name/names and "x" to indicate this tube as the extraction blank for that set. Add 60µl of solution 1 and [REDACTED] to each tube. Add the sample as described in section 5.1 into the bead tube.

5.2.2 Place tubes onto [REDACTED] at maximum speed for 10 minutes.

5.2.3 While tubes are vortexing, label 1 spin filter tube for each sample as well as the blank and 4- 1.5ml tubes for each sample and blank with sample name and number. To the first 1.5ml tube add 250µl of [REDACTED]. To the second 1.5ml tube add 200µl of [REDACTED]. To the third 1.5ml tube add 1200µl of [REDACTED]. Leave the final 1.5ml tube empty and be sure that it is labeled legibly with the entire sample name as this is the final tube in the process and will be used for long term storage.

5.2.4 After the 2ml bead tubes have vortexed for 10 minutes, place them in the centrifuge at 10,000 g for 1 minute.

5.2.5 Transfer the supernatant to the 1.5ml tube containing solution C2. Invert 4-5 times and place at 4°C for 5 minutes.

5.2.6 Place tubes in the centrifuge at 10,000 g at room temperature for 1 minute.

5.2.7 Transfer the supernatant to the 1.5ml tube containing [REDACTED] being sure not to disturb the pellet. Invert 4-5 times and place at 4°C for 5 minutes.

5.2.8 Place tubes in the centrifuge at 10,000g at room temperature for 1 minute.

5.2.9 Transfer 750µls of this to the 1.5ml tube containing [REDACTED]. Invert 4-5 times to mix. Transfer 675µls of this to the spin filter tubes.

5.2.10 Spin at 10,000g for 1 minute. Discard flow through and repeat until all of this solution is used.

5.2.11 After all solution has passed through the spin filter, discard the final flow through. Add 500µls of [REDACTED] directly to the filter. Discard flow through and centrifuge at 10,000g for 1 minute.

5.2.12 Transfer the filter to the remaining clean, labeled 1.5ml tube. Add 100µls of [REDACTED] directly to the filter. Spin at 10,000g for 1 minute. Check tube to be sure that there are 100µls of eluted DNA then discard the filter. Store the extracted DNA at 4°C for up to 6 months. Transfer to -20°C for long term storage.

## **6.0 QUALITY CONTROL**

6.1 For quality control of the extraction process, a recovery standard is added to each sample. An extraction blank is also processed with each extraction set to ensure no cross contamination of samples or solutions.

6.2 Ongoing calibration checks and maintenance of the instruments must be documented in the appropriate logbook.

6.3 The Laboratory Director has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience to process the samples.

6.4 All deviations from this SOP must be documented in a Nonconformance Report (NCR) or an equivalent system (i.e., database).

## DNA Extraction Procedure

Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

SOP No. DNA-EXT

Revision No. 1.0 FINAL

Revision Date: 1/05/06

Effective Date: 1/05/06

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### 7.0 LABORATORY SAMPLE FOLDERS

Extraction volumes for each sample will be logged in at the time of extraction in the worksheet attached in each sample folder. An example of this worksheet is presented in *Attachment 1*.

### 8.0 DOCUMENT CORRECTIONS

Changes or corrections on any laboratory documentation will be made by crossing out the erroneous item with a single line and writing the new information above the crossed-out item. The person correcting the information should initial and date the correction. The original entry, although erroneous, must remain legible beneath the cross-out line. All information will be recorded using black or blue indelible ink.

DNA Extraction Procedure

Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

SOP No. DNA-EXT

Revision No. 1.0 FINAL

Revision Date: 1/05/06

Effective Date: 1/05/06

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## ATTACHMENT 1

### LABORATORY WORKSHEET

## STANDARD OPERATING PROCEDURE

### Quantitative Polymerase Chain Reaction (qPCR)

**SOP Number:** DNA-qPCR

**Revision Number:** 1.0 FINAL

**Revision Date:** 01/10/06

**Effective Date:** 01/10/06

Reviewed By: \_\_\_\_\_ Date \_\_\_\_\_  
Dora Ogles  
DNA Lab Director

Reviewed By: \_\_\_\_\_ Date \_\_\_\_\_  
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QA Coordinator

Reviewed By: \_\_\_\_\_ Date \_\_\_\_\_  
Greg Davis  
President/Lab Director

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## PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the activities involved in the preparation, handling, documentation, and analysis of samples collected from a variety of matrices (water, soil, bio-traps, etc) to enumerate target populations using quantitative Real-Time polymerase chain reaction or qPCR.

## 1.0 SCOPE AND APPLICATION

Quantitative Real-Time polymerase chain reaction (qPCR) is used to enumerate target populations of microorganisms. Microbial Insights, Inc. (MI) uses qPCR to target the following organisms associated with the remediation of a priority pollutants such as Tetrachloroethene (PCE) and Methyl *tert*-butyl ether (MTBE).

Q-Target	MI Code or CAS Number	Real-time assay
<i>Dehalococcoides spp.</i>	qDHC	Taqman
<i>Dehalobacter spp.</i>	qDHB	SYBR green
<i>Desulfuromonas spp.</i>	qDSM	Taqman
<i>Desulfitobacterium spp.</i>	qDSB	SYBR green
Methanogen (mcrA gene)	qMGN	SYBR green
Iron and Sulfate Reducing Bacteria	qIRBSRB	Taqman
Dissimilatory Sulfite Reductase	qDSR	SYBR green
Methane Oxidizing Bacteria	qMOB	SYBR green
PM1	qPM1	Taqman
Universal Baceteria	qEBAC	Taqman
Soluble Methane Monooxygenase	qsMMO	SYBR green
Ammonia Oxidizing Bacteria	qAOB	Taqman
Anaerobic Toluene	qbssA	Taqman
<i>Geobacter spp.</i>	qGEO	Taqman
Denitrifying Bacteria (nirS and nirK)	qDEN	SYBR green
Catechol Dioxygenase	qCAT	SYBR green
Toluene Monooxygenase	qRDEG	SYBR green
Toluene Monooxygenase	qRMO	SYBR green
Xylene Monooxygenase	qTOL	Taqman
Naphthalene Dioxygenase	qNAH	Taqman
Phenol Monooxygenase	qPHE	SYBR green
Acetogens(FTHFS gene)	qACE	SYBR green
Alkane Monooxygenase	qALKb	Taqman
Butane Monooxygenase	qBMO	Taqman
Toluene Dioxygenase	qTOD	Taqman

## 2.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

Samples are collected following the standard operating procedure developed by Microbial Insights, Inc (MI). DNA extraction and RNA extraction are performed following the extraction standard operating procedure developed by Microbial Insights, Inc. DNA is stored in 1/10 Tris-EDTA(TE) at 4°C while RNA is stored in RNA Storage solution at -20°C. RNA samples are converted to cDNA following (SOP-RNA ext) prior to the quantitative PCR analysis.

## 3.0 EQUIPMENT AND SUPPLIES

- 96-well plate (Applied Biosystems).
- Micropipettes (Gilson P-10, P-100, P-1000) and 10 ml pipettes, and appropriate sterile tips (ART). [see calibration listed in calibration log]
- Applied Biosystems SDS 7000 or 7300 instrument. [see calibration listed in calibration log]
- Sterile 0.5ml thin wall strip tubes and optical clear caps (Applied Biosystems)

## 4.0 REAGENTS AND POSITIVE CONTROLS

- 4.1 TaqMan DNA polymerase and master mix for Taqman assays (Applied Biosystems)
- 4.2 Sterile filtered (0.2mM) nanopure, organic-free, deionized water.
- 4.3 Forward and Reverse primers/Probe: these are gene/assay specific (IDT Technologies primers, Applied Biosystems FAM/TAMRA probes). See notes section for primer handling. Final concentration of probe and primers: [REDACTED] or adjusted as needed for each primer set.  
[REDACTED]
- 4.4 Positive and Negative Control DNA: DNA is purchased from ATCC or extracted and purified in-house from pure isolates. The amount of DNA used in all assays is 3µl. For negative controls, water is added in place of template DNA.
- 4.5 [REDACTED] for SYBR green assays ([REDACTED])
- 4.6 [REDACTED] for SYBR green assay
- 4.7 [REDACTED]  
[REDACTED]  
[REDACTED]

## 5.0 PROCEDURE

## 5.1 Preparation of Stock Primer

5.1.1 It is necessary to resuspend and dilute our primers (IDT Technologies) before we can use them for our PCR reaction. This procedure is done when new primers are received and both working stocks and solution stocks should be prepared at this time. The label on the tube will provide the oligo length and concentration.

5.1.2 To the stock tube, aseptically add 1ml of sterile PCR dH<sub>2</sub>O and vortex the tube for 1 minute to resuspend the primer. Centrifuge for 1 minute at 14,000 rpm to remove any solution from the lid then allow the tube to sit at room temperature for 5 minutes to ensure complete dissolution. This provides the solution stock.

5.1.3 To make the working stock to be used in each reaction, add the amount of nanomoles of the stock solution (listed on the stock tube) to a clean 1.5ml tube and bring this volume up to 1ml with sterile dH<sub>2</sub>O.

## 5.2 Mixing Reagents-Taqman Assays

5.2.1 Prepare a master mix including the appropriate amounts of each of the following: PCR water, Taqman Universal PCR mix, Primers (forward and reverse) and Probe. The appropriate amounts are listed in the MI primer list (primer.xls) prepared for each primer/probe combination.

5.2.2 Add [REDACTED] to each well of the 96 well plate or strip tube. Add [REDACTED] template DNA/cDNA to the appropriate wells. Add [REDACTED] positive control DNA to the appropriate wells. Add [REDACTED] PCR water to the negative control wells.

5.2.1 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.2.2 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see table1).

5.2.3 Upon completion of the run, export the data to an Excel worksheet to determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules.

5.2.4 Standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the R<sup>2</sup> of each equation must be at least 0.95.

## 5.3 Mixing Reagents-SYBR green Assays

5.3.1 Prepare a master mix including the appropriate amounts of each of the following ingredients: PCR water, [REDACTED], PCR Primers (forward and reverse), [REDACTED] Reaction Buffer, [REDACTED].  
[REDACTED] The appropriate amounts are listed in the excel sheets prepared for each primer combination.

5.3.2 Add [REDACTED] of master mix to each well of the 96 well plate or strip tube. Add [REDACTED] template DNA/cDNA to the appropriate wells. Add [REDACTED] positive control DNA to the appropriate wells. Add [REDACTED] PCR water to the negative control wells.

5.3.3 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.3.4 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see DNA primers xls data sheet)

5.3.5 Upon completion of the run, export the data to an Excel worksheet, then determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules. The standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the  $R^2$  of each equation must be at least 0.95.

## 6.0 QUALITY CONTROL

6.1 For quality control of the amplification process, positive and negative controls are always performed. The extraction blank from each set is also amplified to ensure no cross contamination of samples or solutions during the extraction process.

6.2 Ongoing calibration checks and maintenance of the instruments must be documented in the appropriate logbook.

6.3 The Laboratory Director has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience to process the samples.

6.4 All deviations from this SOP must be documented in a Nonconformance Report (NCR) or using an equivalent system (i.e., database).

## 7.0 ANALYTICAL REPORT

7.1 All data regarding quantities are entered into a data report using the laboratory information management systems (LIMS).

Quantitative Polymerase Chain Reaction  
Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

SOP No. DNA-qPCR  
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7.2 All documentation pertinent to a project will be placed in the "project folder" identified with the unique LIMS Project number. Hardcopy records, data sheets, and soft copy reports will be stored for a period of three years.

## **8.0 DOCUMENT CORRECTIONS**

Changes or corrections on any laboratory documentation will be made by crossing out the erroneous item with a single line and initialing (by the person performing the correction) and dating the correction. The original item, although erroneous, must remain legible beneath the cross-out line. The new information should be written clearly above the crossed-out erroneous item. All information will be recorded using black or blue indelible ink.

## **9.0 TABLES, DIAGRAMS, AND FLOWCHARTS**

See DNA primer xls sheet for primer conditions.

## STANDARD OPERATING PROCEDURE

### Waste Disposal

**SOP Number:** MI SOP-Waste Disposal

**Revision Number:** 1.0 FINAL

**Effective Date:** 03/01/08

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## 1.0 PURPOSE

This purpose of this standard operating procedure is to provide compliance with the Federal Resource Conservation and Recovery Act (RCRA) in order to identify, handle, store, treat and ultimately dispose of laboratory waste. This waste includes any process waste, unused sample aliquots, and all other laboratory hazardous waste.

## 2.0 RESPONSIBILITY

### 2.1 Chemical Hygiene and Safety Officer

This is the employee authorized to handle the hazardous waste and is responsible for labeling and moving all containers of hazardous waste in the central storage area. He/she is responsible for tracking all hazardous waste in the laboratory and determining and documenting monthly accumulation amounts.

### 2.2 Microbial Insights Director

The Director will oversee all operations and sign all hazardous waste manifests.

### 2.3 Microbial Insights Assistant Director

The assistant Director shall assess any laboratory waste disposal reports and sign hazardous waste manifests in the absence of the Director.

### 2.4 Individual Laboratory Directors and Laboratory Personnel

Laboratory personnel are responsible for segregating hazardous samples at the direction of the chemical hygiene and safety officer.

## 3.0 DEFINITIONS

Hazardous wastes are classified according to type and characteristic. In order for hazardous waste to be handled safely and according to regulations, all generated waste must be classified using the EPA hazardous waste codes from 40 CFR 261. There are two types of Hazardous Waste according to the Resource Conservation and Recovery Act (RCRA). They are: listed waste and characteristic waste.

### 3.1 Listed Waste

There are four classifications of listed wastes (listed in 40 CFR 261):

**F Listed Waste:** Hazardous wastes from non-specific sources such as halogenated solvents used in degreasing. These wastes are assigned a hazardous waste number by the EPA beginning with the letter F. (40 CFR 261.31)

**K Listed Waste:** Hazardous wastes from specific sources such as wastewater treatment sludge from the production of zinc yellow pigments. These wastes are assigned a hazardous waste number by the EPA beginning with the letter K. (40 CFR 261.32)

**P Listed Waste:** Acutely hazardous discarded commercial chemical products, off-specification species, container residues, and spill residues thereof such as an expired container of potassium chloride. These wastes are assigned a hazardous waste number by the EPA beginning with the letter P (40 CFR 261.33)

**U Listed Waste:** Toxic hazardous discarded commercial chemical products, off-specification species, container residues, and spill residues thereof such as an expired container of methylene chloride. These wastes are assigned a hazardous waste number by the EPA beginning with the letter U. (40 CFR 261.33)

### 3.2 Characteristic Waste

Wastes that are classified due to their hazard characteristics are classified using the letter D. Characteristic hazardous wastes are defined as follows:

**Ignitability:** a waste is hazardous if a sample has any of the following properties:

(1) a liquid that has a flash point less than 140° F (60C); (2) not a liquid and is capable, under Standard Temperature and Pressure (STP) of causing fire through friction, absorption of moisture or spontaneous chemical changes and when ignited, burns so vigorously and persistently that it creates a hazard; (3) an ignitable compressed gas; or (4) an oxidizer. These wastes have the EPA Hazardous Waste Number D001.

**Corrosivity:** a liquid that has a pH less than or equal to 2 or greater than or equal to 12.5. These wastes have the EPA Hazardous Waste Number D002.

**Reactivity:** a waste is hazardous due to the characteristic of reactivity if a substance has any of the following properties: (1) is normally unstable and readily undergoes violent change without detonating; (2) reacts violently with water; (3) forms potentially explosive mixtures with water; (4) when mixed with water, generates toxic gases; (5) a cyanide or sulfide bearing waste; (6) capable of detonation if subjected to a strong initiating source or heated under confinement; (7) capable of detonation or explosive decomposition or reaction at STP; or (8) it is forbidden, class A or B explosive according to the DOT. These wastes have the EPA Hazardous Waste Number of D003.

**Toxicity:** If an extract from a sample of the waste meets or exceeds the concentrations listed in the 40 CFR Table 1 of 261.24, the waste is considered hazardous due to the characteristic of toxicity. These wastes have the EPA Hazardous Waste Numbers of D004 through D043.

**Accumulation Date:** The date that the first drop of waste gets placed in a storage container in a hazardous waste storage area.

### 3.3 Waste Disposal Handbook

- 3.3.1 Microbial Insights Waste Disposal Handbook Manual will be kept up to date by the Chemical and Hygiene Safety Officer.
- 3.3.2 The Microbial Insights Director will do a quarterly review of the handbook to be sure that all documents listed in section 3.3.3 are placed into the handbook.
- 3.3.3 The handbook will be located in the hazard waste storage area and will contain:

- 3.3.3.1 Microbial Insights Standard Operating Procedure for Waste Disposal
- 3.3.3.2 Up to date waste disposal information from TDEC
- 3.3.3.3 Records for the past three years including laboratory drum hazardous waste results, appropriately signed manifest documents and the monthly Hazardous Waste Inspection Forms.

## 4.0 WASTE

### 4.1 Inventory control

Chemicals shall be ordered in the smallest quantities possible. It is important that the reagents are used up prior to their expiration date. In most cases, the reagents should be used up before a newer container is opened. This procedure will virtually eliminate the expense of disposing of expired chemicals.

### 4.2 Waste Designations

4.2.1 LIQUID AND SOLID WASTE ARE **NEVER** TO BE PLACED INTO THE SAME DRUM.

4.2.2 Four different waste drums will be maintained for sample disposal (for definitions see 4.2.3). Satellite drums will be maintained in the lipid laboratory labeled with the same designations.

Liquids--Solvent Waste, Filtered Water, Unfiltered Water

Solids—Hazardous Waste Soil

4.2.3 Liquid waste will be immediately placed into waste drums or into waste satellite drums upon completion of a project. Solvent waste will be placed into the appropriately designated 30 gallon drum ("Solvent waste"). Filtered water will be placed into the designated 55 gallon drum ("Filtered water") and unfiltered water and residual sample waste will be placed into the 55 gallon drum designated "Unfiltered water".

#### 4.2.3.1 LIQUID WASTE

4.2.3.1.1 Solvent Waste—only organic solvents used in the Lipid extraction process or pure chloroform, acetone or methanol.

4.2.3.1.2 Filtered Water—only groundwater samples which have been filtered will be placed into this drum.

4.2.3.1.3 Unfiltered Water—extra unfiltered groundwater samples, VFA samples, other liquid waste will be placed into this drum.

#### 4.2.3.2 SOLID WASTE

4.2.3.2.1 Solid Hazardous Waste Drum—any solid material that is known to have contaminant concentrations above those on the TDEC list.

- 4.2.3.2.2 Sorbant materials, gloves and other materials used in hazmat spill cleanups are to be decontaminated after use if possible. Those items that cannot be decontaminated shall be disposed of as hazardous waste.

## 5.0 HAZARDOUS WASTE IDENTIFICATION

### 5.1 "Filtered water" and "Unfiltered Water" Waste Drums

- 5.1.1 Once the "Filtered water" or "Unfiltered Water" drum is full, the following will occur:
- 5.1.2 The person to dispose of the waste will notify the safety officer that a drum is full. A sign will be placed on the drum indicating that it is full and the date.
- 5.1.3 Within seven days, the Chemical Hygiene and Safety Officer will take a sample from the length of the entire drum using the drum sampling device. The sample will be placed into a 1 liter container and this will be repeated until 500mLs have been collected. The 1 liter container will then be inverted at least three times to ensure that the sample is well mixed. Aliquots will be made into four 40mL VOA vials and one 250mL container. A LIMS identifier will be given to the individual drum. The drum as well as sample containers will also be labeled with this identifier.
- 5.1.4 Customer service will ship VOA vials and 250mL container overnight on ice to an accredited laboratory (i.e Microseeps, Inc). The analysis requested on their chain of custody will be 8260 and RCRA metals. Report information will be sent to the Microbial Insights Director (Greg Davis).
- 5.1.5 A pH will be taken of each drum and recorded into the pH excel sheet located in the Waste Disposal file on the server computer (\\Newman\data\Lab Information\Waste Disposal).
- 5.1.6 Once the analytical report is received, the Microbial Insights Director will compare the data with the toxicity limits listed on the TDEC website (also in the Waste Disposal Manual). If any analyte exceeds the listed value, the associated code designation for that chemical or metal will be listed on the drum. This designates the drum as Hazardous Waste. A hazardous waste sticker will be placed onto the drum and filled out with the appropriate designation waste codes. The appropriate waste stream number will be placed onto the label dependent on the contaminants that are elevated in the report. If no analyte exceeds the listed values, a non-hazardous sticker will be placed onto the drum.
- 5.1.7 A copy of the analytical report will be placed into the appropriate year of the Microbial Insights Waste Disposal Handbook. The original report copy will be placed into the office storage file (see Sandy White).

- 5.1.8 When two full hazardous waste drums are on site, a call will be placed to the waste disposal company (i.e. Safety Kleen) for pick-up. No more than two full 55 gallon hazardous waste drums will be on site at any given time.
- 5.1.9 If a non-hazardous waste sticker is placed on the drum, no immediate pick up is required. The drum can be picked up in the future when hazardous drums are taken. No more than four 55 gallon non-hazardous drums will be on site at any given time.

## 5.2 Solvent Waste Drum

- 5.2.1 Once a "Solvent Waste" drum is full, the following will occur:
- 5.2.2 The person to dispose of the waste will notify the safety officer that a drum is full. A sign will be placed on the drum indicating that it is full and the date.
- 5.2.3 A hazardous sticker will be placed onto the drum with codes D001, D022 and F005. Waste stream 1 will also be listed on the drum.
- 5.2.4 When two full hazardous waste drums are on site, a call will be placed to the waste disposal company (i.e. Safety Kleen) for pick-up. No more than two full 55 gallon hazardous waste drums will be on site at any given time.

## 5.3 Solid Waste

- 5.3.1 Any solid waste such as soil will be handled as follows:
- 5.3.2 Soil waste will be dried by either:
  - 5.3.2.1 Placing into the fume hood and left open to dry with the hood left on.
  - 5.3.2.2 Placing into the lyophilizer and dried.
- 5.3.3 Once dry, soil will be placed into an autoclave bag inside a 5 gallon satellite container located in the Lipid Laboratory fume hood. No more than 20 pounds of soil waste will be accumulated before a composite "Soil Satellite Test Sample" will be tested.
- 5.3.4 When no more than 20 pounds of soil waste have accumulated, the Chemical Hygiene and Safety Officer will take an 8oz. composite sample and label it as "Soil Satellite Test Sample". It will be then given to customer service and the sample will be given an MI Identifier and shipped overnight on ice to an accredited laboratory (i.e Microseeps, Inc) for TCLP and RCRA metal testing (listed as such on their chain of custody). The same MI Identifier will be placed onto the autoclave bag containing the soil waste being tested.
- 5.3.5 Results will be reported to the Microbial Insights Director and compared with the toxicity limits listed on the TDEC website (also in the Waste Disposal Manual). If any analyte exceeds the listed value, the autoclave bag containing that waste will be transferred into a 30 gallon drum designated "Hazardous Waste Soil Drum". A hazardous waste sticker will be placed onto the drum and filled out with the

appropriate designation waste codes. Each time solid material is added to the drum, the appropriate codes will be placed onto the label. The appropriate waste stream number will be placed onto the label dependent on the contaminants that are elevated in the report.

- 5.3.6 A copy of the analytical report will be placed into the appropriate year of the Microbial Insights Waste Disposal Handbook. The original report copy will be placed into the office storage file (see Sandy White).
- 5.3.7 When two full hazardous waste drums are on site, a call will be placed to the waste disposal company (i.e. Safety Kleen) for pick-up. No more than two full 55 gallon hazardous waste drums will be on site at any given time.
- 5.3.8 If no analyte exceeds the listed values, the autoclave bag containing the tested soil will have autoclave tape affixed to the bag. This bag will be autoclaved and placed into the dumpster.

#### 5.4 Unused, Expired, or Off Specification Laboratory Chemicals

- 5.4.1 When a hazardous chemical or reagent can no longer be used because it has expired or there is a question about its purity, the Chemical Hygiene and Safety Officer shall be notified. The Chemical Hygiene and Safety Officer shall see that the chemical is labeled with all applicable EPA Hazardous Waste Numbers and the accumulation date.
- 5.4.2 The Chemical Hygiene and Safety Officer shall remove the chemical from the laboratory, store it in a hazardous waste storage area, and include it in the next scheduled waste pick up.

## 6.0 HAZARDOUS WASTE HANDLING

### 6.1 Administrative Controls

- 6.1.1 Exposure to hazardous waste will be controlled by limiting access to only designated individuals who have specific responsibilities concerning waste.
- 6.1.2 The handling of large volumes of hazardous waste (30 or 55 gallon drums) will be handled only by the Chemical Hygiene and Safety Officer. Thus limiting the exposure of other individuals.
- 6.1.3 Each laboratory will have a designated spot for accumulating waste and that waste will be removed as soon as the waste container is full.
- 6.1.4 Each quarter, the Chemical Hygiene and Safety Officer will do a walkthrough of all laboratories where hazardous waste is handled and document any concerns. These concerns will be brought to the individual Laboratory Directors and if necessary, the Microbial Insights Director.

### 6.2 Engineering Controls

- 6.2.1 Hazardous waste that poses a hazard by inhalation shall be handled under a fume hood with the sash at the designated height.
- 6.2.2 All hazardous waste shall be stored in properly ventilated cabinets until removed to the hazardous waste storage area.
- 6.2.3 Containers of waste that weigh more than twenty pounds shall be transported or moved by using either a dolly or a wheeled cart.

### 6.3 Personal Protection

- 6.3.1 Personal Protective Equipment must be worn while handling hazardous waste. All employees who are disposing of waste will wear safety glasses and gloves.

## 7.0 HAZARDOUS WASTE STORAGE

### 7.1 Storage Area

- 7.1.1 All waste that is deemed hazardous shall be stored in compatible containers as recommended by the waste hauler.
- 7.1.2 The Chemical Hygiene and Safety Officer will label all waste as described in section 5.0 of this SOP.
- 7.1.3 All hazard warning signs will be posted designating the hazardous waste area.
- 7.1.4 The Chemical Hygiene and Safety Officer will inspect the waste storage area monthly and will immediately report any signs of container deterioration to the MI Laboratory Director. Also any regulatory or safety concerns will be notated (see attached Exhibit 1).

### 7.2 Accumulation Amounts

- 7.2.1 Microbial Insights generates less than 220 lbs of hazardous waste per month and is designated as a conditionally exempt small quantity generator (CESQG).
- 7.2.2 Total hazardous waste accumulation shall never exceed 2000 lbs on site.

## 8.0 WASTE DISPOSAL

All hazardous waste shall be disposed in accordance with the regulations outlined in 40 CFR Part 262 Subpart B-The Manifest. The contracted waste hauler will complete the manifest and submit it to the Chemical Hygiene and Safety Officer for inspection and signature. It is the Chemical Hygiene and Safety Officer's responsibility to ensure that the manifest is complete and correct.

### 8.1 Manifest Documentation

- 8.1.1 The Chemical Hygiene and Safety Officer shall do the following:
  - 8.1.1.1 Obtain the handwritten signature of the Microbial Insights Director

- 8.1.1.2 Obtain the signature of the initial transporter and the date of acceptance on the manifest.
- 8.1.1.3 Retain one copy of the manifest.
- 8.1.1.4 Give the transporter the remaining copies.
- 8.1.1.5 Make a copy of the manifest document. Give the original manifest to the Microbial Insights Director for filing. The copy should be placed into the Hazardous Waste Disposal Handbook. The original should be placed in the office storage file. All original copies will be stored for at least three years.

When packaged for transportation and disposal, the contracted waste hauler will label the container with the proper Department of Transportation label and insure that the package is prepared for Transportation in accordance with 49 CFR.

## 9.0 REFERENCES

- 9.1 40 Code of Federal Regulations. Protection of Environment, Parts 260 to 299. Office of the Federal Register National Archives and Records Administration.

Exhibit 1

**Hazardous Waste Storage Area Inspection Form**

**Date:** \_\_\_\_\_

Is the waste contained in appropriate drums?	Yes	No
Is the area marked with hazard warning signs?	Yes	No
Are containers of waste capped?	Yes	No
Is the storage log up to date?	Yes	No
Are containers labeled?	Yes	No
Do any containers show signs of deterioration?	Yes	No
How much waste (in pounds) do we currently have?	Yes	No
Do we have any acutely hazardous waste?	Yes	No
If so, how much? _____		

Any other items of regulatory or safety concerns that should be addressed?

\_\_\_\_\_

\_\_\_\_\_

Inspector's Signature: \_\_\_\_\_

Printed Name: \_\_\_\_\_

MI Laboratory Director Signature: \_\_\_\_\_

**KB LABS, INC.**

**Sample Receipt and Acceptance**

- 1.0 KB Labs will supply pre-cleaned and certified sample containers to the client as required.
- 2.0 The Field Chemist will initiate and release to the client a Chain-of-Custody (COC) Record at the time the sample containers are released.
- 3.0 The Field Chemist will receive samples from a member of the client field sampling team and is the designated sample custodian.
- 4.0 When samples are received, the Field Chemist will determine the following:
  - 4.1 Whether samples have been received on ice.
  - 4.2 Whether the samples are in the appropriate sample containers. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.3 Whether the samples are of adequate volume or mass.
  - 4.4 Whether there are signs of leakage, breakage, or contamination.
  - 4.5 Whether headspace is present in the sample container (VOCs only).
  - 4.6 Whether proper preservation (if any) has been added. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.7 Whether holding times have been met. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.8 Whether samples are properly labeled. Proper labeling will include the following:
    - The use of indelible ink.
    - The use of durable labels that are not easily removed.
    - Writing that can be clearly read and understood.
  - 4.9 Whether the COC Record has all the necessary and proper documentation. This will include the following:
    - Identification of samples
    - Location of sample collection
    - Date and time of sample collection
    - Sample type
    - Any special comments concerning the sample
    - Signatures of field sampler and sample custodian/field chemist

- 4.10 All samples received from the client must be recorded on the COC regardless of what the client says.
- 5.0 If any of the above conditions are not met, the Field Chemist will note the improper conditions for each sample on the COC Record, and he will immediately notify the field team leader of these improper conditions.
- 6.0 The field team leader must then make the decision whether to reject the samples in question.
- 7.0 Only after verifying the status of the samples, the Field Chemist will sign the Chain-of-Custody (COC) Record.
- 8.0 The Field Chemist will place the third copy (pink) of the COC Record into the sample receipt logbook.
- 9.0 The Field Chemist will return the second (yellow) copy to the client field team leader.
- 10.0 The Field Chemist will retain the original (white) copy of the COC Record with the project data file.

**KB LABS, INC.**

**Table 1: Analytical Run Sequence/Surrogate Percent Recoveries**

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

Sample ID	Date of Analysis	Surrogate % Recovery				Surrogate Control Limits			
		S1*	S2*	S3*	S4*	S1*	S2*	S3*	S4*
Example		99	102	105	98	Pass	Pass	Pass	Pass
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
<b>Comments:</b>		Although some surrogates may be out of the control percent recovery range, other supporting QC, such as matrix spikes, matrix spike duplicates, method blanks, and laboratory control samples, are performed by KB Labs to further validate reported data.							

**\*Surrogate Compounds:**

S1 = Dibromofluoromethane (54% - 149%)

S2 = 1,2- Dichloroethane-D4 (61% - 156%)

S3 = Toluene-D8 (72% - 127%)

S4 = 4-Bromofluorobenzene (72% - 125%)

**KB LABS, INC.**

**Table 2: VOC Spike Compound Percent Recoveries**

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No.:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

**Matrix Spike/Matrix Spike Duplicate (MS/MSD):**

Matrix Spike Compounds	Date of Analysis:								
	Control Limits			Percent Recoveries			Control Limit Checks		
	Lower	Upper	RPD	MS	MSD	RPD	MS	MSD	RPD
Dichlorodifluoromethane	30	163	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dichloromonofluoromethane	53	156	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloromethane	34	142	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Vinyl Chloride	40	143	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromomethane	30	147	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloroethane	30	160	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Trichlorofluoromethane	45	154	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Freon 113	50	143	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1-Dichloroethene	45	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Methylene Chloride	49	148	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
trans-1,2-Dichloroethene	47	151	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
MtBE	41	153	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1-Dichloroethane	51	142	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
2,2-Dichloropropane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
cis-1,2-Dichloroethene	59	156	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloroform	65	139	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Carbon Tetrachloride	47	150	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Benzene	65	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Trichloroethene	70	136	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Toluene	79	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,1-Trichloroethane	54	146	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichloroethane	71	147	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichloropropane	64	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dibromomethane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromodichloromethane	50	166	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
c-1,3-Dichloropropene	52	168	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
t-1,3-Dichloropropene	71	152	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,2-Trichloroethane	61	150	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dibromoethane	59	157	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dibromochloromethane	58	157	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chlorobenzene	79	121	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,1,2-Tetrachloroethane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Tetrachloroethene	57	152	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3-Dichloropropane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Ethylbenzene	74	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
m,p-Xylene	73	132	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
o-Xylene	74	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Styrene	74	123	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromoform	42	149	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Isopropylbenzene	76	131	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,2,2-Tetrachloroethane	53	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromobenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
n-Propylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
2-Chlorotoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
4-Chlorotoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3,5-Trimethylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
tert-Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,4 Trimethylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
sec Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
p-Isopropyltoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3-Dichlorobenzene	77	122	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,4-Dichlorobenzene	79	120	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichlorobenzene	78	121	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
n-Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dibromo-3-chloropropan	36	161	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,4-Trichlorobenzene	57	154	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Hexachlorobutadiene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Naphthalene	47	158	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,3-Trichlorobenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!

**Note:** Control Limits are based on a semi-annual historical evaluation of mobile unit.

**KB LABS, INC.**

**Table 2: VOC Spike Compound Percent Recoveries**

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No.:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

**Laboratory Control Spikes (LCS):**

Spike Compounds	Control Limits		Percent Recoveries			Control Limit Checks		
	Lower	Upper	LCS#1	LCS#2	LCS#3	LCS#1	LCS#2	LCS#3
	Dichlorodifluoromethane	43	to 178	0	0	0	< LCL	< LCL
Chloromethane	42	to 176	0	0	0	< LCL	< LCL	< LCL
Vinyl Chloride	28	to 161	0	0	0	< LCL	< LCL	< LCL
Bromomethane	31	to 157	0	0	0	< LCL	< LCL	< LCL
Chloroethane	44	to 162	0	0	0	< LCL	< LCL	< LCL
Trichlorofluoromethane	46	to 167	0	0	0	< LCL	< LCL	< LCL
Freon 113	59	to 168	0	0	0	< LCL	< LCL	< LCL
1,1-Dichloroethene	43	to 156	0	0	0	< LCL	< LCL	< LCL
Methylene Chloride	43	to 160	0	0	0	< LCL	< LCL	< LCL
trans-1,2-Dichloroethene	39	to 169	0	0	0	< LCL	< LCL	< LCL
MtBE	46	to 157	0	0	0	< LCL	< LCL	< LCL
1,1-Dichloroethane	43	to 156	0	0	0	< LCL	< LCL	< LCL
2,2-Dichloropropane	70	to 130	0	0	0	< LCL	< LCL	< LCL
cis-1,2-Dichloroethene	60	to 150	0	0	0	< LCL	< LCL	< LCL
Chloroform	60	to 145	0	0	0	< LCL	< LCL	< LCL
Carbon Tetrachloride	43	to 154	0	0	0	< LCL	< LCL	< LCL
Benzene	63	to 129	0	0	0	< LCL	< LCL	< LCL
Trichloroethene	47	to 164	0	0	0	< LCL	< LCL	< LCL
Toluene	78	to 128	0	0	0	< LCL	< LCL	< LCL
1,1,1-Trichloroethane	52	to 152	0	0	0	< LCL	< LCL	< LCL
1,2-Dichloroethane	68	to 147	0	0	0	< LCL	< LCL	< LCL
1,2-Dichloropropane	53	to 159	0	0	0	< LCL	< LCL	< LCL
Dibromomethane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Bromodichloromethane	45	to 170	0	0	0	< LCL	< LCL	< LCL
c-1,3-Dichloropropene	50	to 174	0	0	0	< LCL	< LCL	< LCL
t-1,3-Dichloropropene	78	to 167	0	0	0	< LCL	< LCL	< LCL
1,1,2-Trichloroethane	67	to 149	0	0	0	< LCL	< LCL	< LCL
1,2-Dibromoethane	65	to 153	0	0	0	< LCL	< LCL	< LCL
Dibromochloromethane	68	to 151	0	0	0	< LCL	< LCL	< LCL
Chlorobenzene	79	to 128	0	0	0	< LCL	< LCL	< LCL
1,1,1,2-Tetrachloroethane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Tetrachloroethene	60	to 151	0	0	0	< LCL	< LCL	< LCL
1,3-Dichloropropane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Ethylbenzene	77	to 125	0	0	0	< LCL	< LCL	< LCL
m,p-Xylene	77	to 134	0	0	0	< LCL	< LCL	< LCL
o-Xylene	79	to 125	0	0	0	< LCL	< LCL	< LCL
Styrene	76	to 124	0	0	0	< LCL	< LCL	< LCL
Bromoform	52	to 148	0	0	0	< LCL	< LCL	< LCL
Isopropylbenzene	87	to 138	0	0	0	< LCL	< LCL	< LCL
1,1,2,2-Tetrachloroethane	59	to 143	0	0	0	< LCL	< LCL	< LCL
Bromobenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
n-Propylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
2-Chlorotoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
4-Chlorotoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,3,5-Trimethylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
tert-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2,4-Trimethylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
sec-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
p-Isopropyltoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,3-Dichlorobenzene	84	to 117	0	0	0	< LCL	< LCL	< LCL
1,4-Dichlorobenzene	84	to 122	0	0	0	< LCL	< LCL	< LCL
1,2-Dichlorobenzene	85	to 116	0	0	0	< LCL	< LCL	< LCL
n-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2-Dibromo-3-chloropropane	43	to 169	0	0	0	< LCL	< LCL	< LCL
1,2,4-Trichlorobenzene	63	to 149	0	0	0	< LCL	< LCL	< LCL
Naphthalene	52	to 150	0	0	0	< LCL	< LCL	< LCL
Hexachlorobutadiene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2,3-Trichlorobenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL

**Note:** Control limits are based on method guidance.





State of Florida  
Department of Health, Bureau of Laboratories  
This is to certify that  
E82816

K B LABS, INC.; KB-3  
25132 SW 1ST AVENUE AMERICA'S BODY COMPANY S/N 10959  
(MOBILE LAB)  
NEWBERRY, FL 32669

has complied with Florida Administrative Code 64E-1,  
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

**EFFECTIVE July 01, 2009 THROUGH June 30, 2010**



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 E82816-12-07/01/2009  
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 4

Attachment to Certificate #: E82816-12, expiration date June 30, 2010. This listing of accredited analytes should be used only when associated with a valid certificate.

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EPA Lab Code: FL01171

(352) 367-0073

E82816

K B Labs, Inc.; KB-3

25132 SW 1st Avenue

America's Body Company S/N 10959 (mobile lab)

Newberry, FL 32669

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Benzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromoform	EPA 8260	Volatile Organics	NELAP	2/7/2008
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroform	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/7/2008

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Issue Date: 7/1/2009

Expiration Date: 6/30/2010



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Newberry, FL 32669

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Styrene	EPA 8260	Volatile Organics	NELAP	2/7/2008
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Toluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/7/2008

Charlie Crist  
Governor



Ana M. Viamonte Ros, M.D., M.P.H.  
State Surgeon General

### Laboratory Scope of Accreditation

Page 3 of 4

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Newberry, FL 32669

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/7/2008
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Benzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Bromoform	EPA 8260	Volatile Organics	NELAP	2/7/2008
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Chloroform	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/7/2008

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Ana M. Viamonte Ros, M.D., M.P.H.  
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Page 4 of 4

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Newberry, FL 32669

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Styrene	EPA 8260	Volatile Organics	NELAP	2/7/2008
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Toluene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/7/2008
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/7/2008
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/7/2008
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/7/2008

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Issue Date: 7/1/2009

Expiration Date: 6/30/2010

KB Labs, Inc.

**ANALYTICAL STANDARD OPERATING  
PROCEDURE No. 1**

**DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY PURGE &  
TRAP GAS CHROMATOGRAPHY/MASS SPECTROMETRY –  
METHOD 8260B**

Signature of Approving Authority: \_\_\_\_\_

Michael G. Winslow  
Quality Assurance Officer

Effective Date: October 2008

**Analytical Standard Operating Procedure No. 1**  
**Revision 4**

**DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY PURGE & TRAP GAS  
CHROMATOGRAPHY/MASS SPECTROMETRY – METHOD 8260B**

**1.0 SCOPE AND APPLICATION**

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water and soil samples by purge and trap/gas chromatography/mass spectrometry.

1.2 The following compounds may be determined by this method:

1,1,1,2-Tetrachloroethane	2-Chlorotoluene	Isopropylbenzene
1,1,1-Trichloroethane	4-Chlorotoluene	m&p-Xylene
1,1,2,2-Tetrachloroethane	Benzene	Methylene chloride
1,1,2-Trichloroethane	Bromobenzene	MtBE
1,1-Dichloroethane	Bromochloromethane	Naphthalene
1,1-Dichloroethene	Bromodichloromethane	n-Butylbenzene
1,1-Dichloropropene	Bromoform	n-Propylbenzene
1,2,3-Trichlorobenzene	Bromomethane	o-Xylene
1,2,3-Trichloropropane	c-1,2-Dichloroethene	p-Isopropyltoluene
1,2,4-Trichlorobenzene	c-1,3-Dichloropropene	sec-Butylbenzene
1,2,4-Trimethylbenzene	Carbon tetrachloride	Styrene
1,2-Dibromo-3-chloropropane	Chlorobenzene	t-1,2-Dichloroethene
1,2-Dibromoethane	Chloroethane	t-1,3-Dichloropropene
1,2-Dichlorobenzene	Chloroform	tert-Butylbenzene
1,2-Dichloroethane	Chloromethane	Tetrachloroethene
1,2-Dichloropropane	Dibromochloromethane	Toluene
1,3,5-Trimethylbenzene	Dibromomethane	Trichloroethene
1,3-Dichlorobenzene	Dichlorodifluoromethane	Trichlorofluoromethane
1,3-Dichloropropane	Ethylbenzene	Vinyl chloride
1,4-Dichlorobenzene	Hexachlorobutadiene	
2,2-Dichloropropane		

**2.0 SUMMARY OF METHOD**

2.1 The VOCs are introduced into the gas chromatograph by the purge-and-trap technique as described in EPA SW 846 Method 5030B for waters and EPA SW846 Method 5035A for soils. Samples are purged with helium and the volatile components are collected on a solid-phase adsorption trap.

2.2 After purging is complete, the adsorption trap is heated and back-purged with helium to desorb the trapped components into a gas chromatograph for separation on a narrow bore capillary column. Components eluted from the capillary column are introduced directly into a mass spectrometer for qualitative and quantitative determination based on EPA SW846 Method 8260B.

2.3 The individual volatile components are measured against appropriate standards. Identification of the target compounds is accomplished by comparing their mass spectra with the electron impact mass spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a five-point calibration curve.

2.4 The estimated quantitation limits (EQL) or reporting limits established by KB Labs for this method are 1 ug/L for low-level water samples and 2 - 10 ug/kg wet-weight for soil samples. The actual limits are dependent upon individual compound purging efficiency, the amount of sample used, and the particular sample matrix.

### **3.0 DEFINITIONS**

Refer to Sec 5.0, Chapter 1, *Test Methods for Evaluating Solid Wastes, Fourth Edition, SW-846*.

### **4.0 INTERFERENCES**

4.1 The analyst must be careful not to introduce major sources of VOC contamination into the laboratory. These sources include organic extraction solvents, impurities in the purging gas and sorbent trap, and the use of non-PTFE sealants, plastic tubing, or flow controllers with rubber components. A method or reagent blank should be analyzed to determine whether contaminants are present. Subtracting blank values from sample results is not permitted.

4.2 Contamination can occur when a sample containing low concentrations of VOCs is analyzed immediately after one containing high concentrations of VOCs. The analyst should rinse the sample transfer syringe with two portions of reagent water after each sample transfer into the autosampler purging chambers. If time allows, reagent water blanks can be placed between samples in the autosampler device.

4.3 To reduce the chances of sample and system contamination, all samples are screened prior to analysis by GC/MS. Screening is performed by analyzing sample headspace using GC/FID.

### **5.0 SAFETY**

Refer to procedures described in KB Labs' *Health and Safety Manual*.

### **6.0 EQUIPMENT AND SUPPLIES**

6.1 Purge-and-trap concentrator: Tekmar Model LSC 3000

6.2 Purge-and-trap autosampler: Varian Arcon

6.3 Gas chromatograph/mass spectrometer (GC/MS) system: Hewlett-Packard (HP) 6890A GC/HP5973A MS / Chem Station

6.4 Gas chromatograph/flame ionization detector: HP 5890A with a HP 3396 integrator.

6.5 GC column for GC/MS: DB624, 20 m x 0.18mm, 1.0mm film thickness

6.6 Syringes: 10 uL, 100 uL, 1 mL (gas tight), and 10 mL,

6.7 Volumetric flasks: 10 mL, 100 mL

6.8 Glass vials: 40 mL with PTFE-lined septum screw caps.

6.9 PTFE-lined screw cap vials: 2 mL

6.10 Disposable pipets: 1 mL Pastuer.

6.11 Top-loading balance: Ohaus SC2020, capable of weighing 0.01 grams.

## **7.0 REAGENTS AND STANDARDS**

7.1 Methanol, purge and trap grade.

7.2 Reagent water: VOC free (determined from method blank analysis)

7.3 Stock Calibration Standard Solutions

7.3.1 Volatiles (54 components): 200 ug/mL in methanol, purchased from Accustandard.

7.3.2 Gases (6 components): 200 ug/mL in methanol, purchased from Accustandard.

7.4 Stock Calibration Verification Standard Solutions (second source)

7.4.1 Volatiles (54 components): 200 ug/mL in methanol, purchased from Restek.

7.4.2 Gases (6 components): 200 ug/mL in methanol, purchased from Restek

7.5 Stock Internal Standard and Surrogate Solutions

7.5.1 7 Components: 2000 ug/mL, purchased from Accustandard.

## 7.6 Preparation of Calibration Standards

7.6.1 A *calibration standard spiking solution* is first prepared by diluting 1 mL of each 200 ug/mL stock calibration standard (Accustandard volatile and gas) to 10 mL with methanol. This working stock has a concentration of 20 ug/mL.

7.6.2 *Calibration standards in reagent water* are then prepared from the working stock solution according to the dilution scheme outlined below:

Volume of Calibration Standard Spiking Solution Added (uL)	Volume of Reagent Water (mL)	Concentration of Calibration Standard (ug/L)
0.5	10	1
2.5	10	5
5	10	10
10	10	20
25	10	50
50	10	100

## 7.7 Preparation of Calibration Verification Standard

7.7.1 A *calibration verification standard spiking solution* is prepared by diluting 1 mL of each stock calibration standard (Restek volatile and gas) to a 10 mL volumetric flask and bringing to volume with methanol. This *calibration verification standard spiking solution* has a concentration of 20 ug/mL.

7.7.2 A *calibration verification standard in water* is prepared by adding either 10 or 25 uL of the calibration verification standard spiking solution to 10 mL of reagent water contained in a 10-mL gas-tight syringe. The concentration of the *calibration verification standard* is 20 ug/L or 50 ug/L.

## 7.8 Preparation of Internal Standards and Surrogate Standards

7.8.1 An intermediate *internal standard/surrogate stock solution* is prepared by diluting 1 mL of the 2000 ug/mL stock standard into 10 mL of methanol. This intermediate stock solution has a concentration of 200 ug/mL.

7.8.2 A *internal standard/surrogate spiking solution* is prepared by adding 1 mL of the intermediate internal standard/surrogate stock solution to 10 mL of methanol. This spiking solution has a concentration of 20 ug/mL.

7.8.3 10 uL of the internal standard/surrogate spiking solution is added to all 10 mL water standards and samples prior to analysis. This will give a 20 ug/L concentration for internal standard and surrogate compounds.

## 7.9 Storage of Standards

7.9.1 All standards will be stored in a freezer @ -10 °C or less.

7.9.2 All unopened stock standards in methanol have an expiration day assigned by the manufacturer.

7.9.3 All standards prepared in methanol will be stored in 2 mL-PTFE-lined screw cap vials without headspace in the vial. Standards with partial headspace in the vial will not be retained.

7.9.4 VOC stock standards in methanol with permanent gases that are stored long term in the office VOC freezer expire one week after opening unless acceptability of the standard can be documented.

7.9.5 VOC standards in methanol with non-gaseous compounds that are stored long-term in the office VOC freezer expire 6 months after opening unless acceptability of the standard can be documented.

7.9.6 Secondary standards in methanol have a one-week holding time.

7.9.7 Vials expire 7 days from when the septum is punctured.

7.9.8 All standards will be stored separately from samples

## 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

### 8.1 Sample Collection

8.1.1 Sample containers are purchased pre-cleaned and with a Certificate of Quality Assurance/Analysis from an approved vendor and will be supplied to the client by KB Labs prior to sampling.

#### 8.1.2 Collection of Water Samples

8.1.2.1 Each water sample is collected in two 40-mL glass vials with open-top screw caps fitted with PTFE-lined septa. The duplicate vial is potentially used for MS/MSD analysis. Once the sample is collected, the septum must be placed with the PTFE side towards the water sample and the open-top cap tightened finger tight.

8.1.2.2 Water samples must be collected without headspace (no bubbles).

#### 8.1.3 Collection of Soil Samples

8.1.3.1 For soil samples, approximately five grams of sample is collected in each of three pre-weighed 40-mL glass vials with open-top screw cap fitted with PTFE-lined septa. The pre-weighed vials contain 10 mL of laboratory reagent water. Two of the samples should be collected for potential duplicate spike samples. Once a sample is placed in a vial, the septum must be placed with the PTFE side towards the soil sample and the open-top cap tightened finger tight.

8.1.3.2 In case high concentrations ( $> 200$  ug/kg) of target analytes are suspected from screening analysis, approximately five grams of sample is collected in a pre-weighed 40-mL glass vial/PTFE-lined septum containing 10 mL of methanol. Once the sample is collected, the septum must be placed with the PTFE side towards the soil sample and the open-top cap tightened finger tight.

8.1.3.3 In order to accommodate for the determination of percent moisture determinations for soil samples, an additional 40-mL vial, filled to the top with the soil sample, should be collected.

8.1.4 The label on each sample container must have a distinguishing field identification for each sample and be accompanied by a properly completed chain-of-custody form. The weight of each prepared vial should be recorded on the label to the nearest .01 grams. Refer to KB Labs' Standard Operating Procedure (SOP) No. 007 for a description of sample receipt and acceptance procedures prior to sample analysis.

## 8.2 Sample Preservation and Storage

8.2.1 Even though samples are generally received and analyzed in the mobile lab soon after collection by the client, they must still be received on ice and preserved with acid to  $\text{pH} < 2$ . (Add 0.12 grams of sodium bisulphate to each vial before – degradation of unsaturated

8.2.2 If a sample is not processed immediately after receipt, it will be stored in an iced cooler at  $4 \pm 2$  °C until ready for processing. The sample should be allowed to come to room temperature before processing for analysis.

8.2.3 Because samples are generally analyzed in the mobile lab the same day as receipt, holding times should not be an issue. The regulatory holding times for water samples refrigerated at  $4 \pm 2$  °C is 7 days and for soil samples 14 days. Soils samples collected in water, if not analyzed within 48 hrs, must be frozen – after freezing they have a 14-day holding time from the time of collection. Samples preserved with sodium bisulfate in the field have a 14-day holding time.

## 9.0 QUALITY CONTROL

### 9.1 Initial Demonstration of Capability (IDOC)

9.1.1 A new analyst will perform an IDOC prior to using any test method for the analysis of client samples.

9.1.2 An IDOC will also be performed whenever a new instrument or method is implemented.

9.1.3 The IDOC will be performed in a clean and applicable matrix.

9.1.5 Four replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest at 10 – 50 times the method detection limits for the analytes.

9.1.6 The concentrations for each analyte are then experimentally determined using the standard operating procedures for the analytical method.

9.1.7 The mean recovery and standard deviation of the found concentrations for the replicates is then calculated for each analyte, and these are then compared to the corresponding acceptance criteria for accuracy and precision established by the lab from historical data. Acceptance criteria established by the lab may not exceed 70 – 130%.

9.2 Quality control procedures for the operation of the GC/MS include:

9.2.1 The GC/MS system must be *tuned* to meet specified BFB criteria described in Section 10.1.

9.2.2 *Initial calibration* of the GC/MS system must be performed as described in Section 10.2.

9.2.3 *Calibration verification* procedures must be performed every 12 hours of instrument operation as described in Section 10.3 and the CCC, SPCC, and IS criteria must be met.

9.2.4 A *laboratory reagent blank (method blank)* must be analyzed in order to monitor the cleanliness of the analytical system. The method blank must be analyzed after the calibration standard(s) and before the samples and the results must demonstrate that the analytical system contains less than 20% of the reporting level for all target compounds and is free of any contaminants that might interfere with the analysis of the target compounds. Method blanks may be analyzed at a higher frequency if deemed necessary by the operator.

9.2.5 All samples, including standards and method blanks, must be fortified with *surrogate* and *internal standards*. The percent recovery of each surrogate compound is calculated in order to evaluate the performance of the analytical system and to help determine the potential for sample matrix effects. Surrogate compounds include 1,2-dichloroethane-d4, 1,4-difluorobenzene, toluene-d8, and 4-bromofluorobenzene. Surrogate control limits are set at  $\pm 3$  standard deviations of the laboratory average historical recoveries. Quantitation is performed

with the internal standards which include pentafluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4.

9.2.6 Duplicate *matrix spike (MS/MSD)* samples must be analyzed for every 20 samples analyzed and for any daily sample batch that is less than 20 samples in order to monitor the performance (precision and accuracy) of the target compounds in the actual matrix. The accuracy (% Recovery) and precision (%RPD) is calculated for every pair of spikes and a statistical analysis is performed on at least the last 20 samples analyzed in order to calculate and update control limits. The matrix spike samples are prepared by spiking 10 mL aliquots of a selected water sample (or 5 gram soil sample) with the appropriate uL amounts of the matrix spiking standard, which are prepared from different stock standards than the calibration standards.

9.2.7 A single *laboratory control sample (LCS)* must be analyzed for every 20 samples analyzed and for any daily sample batch that is less than 20 samples in order to monitor the recovery of the target compounds from a clean sample matrix. A accuracy (% Recovery) is calculated and a statistical analysis is performed on at least the last twenty samples analyzed in order to calculate and update control limits.

## 10.0 ANALYTICAL PROCEDURE

### 10.1 Calibration and Standardization

10.1.1 *Bromofluorobenzene (BFB) tuning* criteria must be met at the beginning of each day and every 12 hours thereafter as long as analyses are performed. The following tuning criteria from EPA Method 8260 must be met before any samples or standards are analyzed.

<u>M/z</u>	<u>Required Intensity (relative abundance)</u>
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

10.1.2 *Initial calibration* is performed when the instrument is started up when the instrument response has drifted out of calibration in order to demonstrate that the instrument is capable of acceptable performance at the beginning of the analytical run and is producing a linear calibration.

10.1.2.1 Five or six calibration standards @ 1,5,10,20,50, and 100 ug/L are analyzed.

10.1.2.2 The percent relative standard deviation (%RSD) of each target analyte must be  $\leq 15\%$ .

10.1.2.3 The system performance check compounds (SPCCs) must pass the following minimum mean response factor (RF) criteria:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

10.1.2.4 The following calibration check compounds (CCCs) must meet minimum RSD criteria of  $\leq 30\%$

1,1-Dichloroethane	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropene	Vinyl chloride

10.1.3 A *calibration verification standard (CVS)* at least every 12 hours in order to verify initial calibration.

10.1.3.1 A 20 ug/L or 50 ug/L standard must be analyzed.

10.1.3.2 The SPCCs must pass the minimum mean RF criteria as in initial calibration.

10.1.3.3 The percent difference of the CCCs must be  $\leq 20\%$  of initial calibration, or if not included in the target compounds, all analytes must be  $\leq 20\%$  of initial calibration.

10.1.3.4 The internal standard (IS) retention times must be  $< 30$  secs from those in the midpoint standard of the most recent initial calibration.

10.1.3.5 The IS responses must be within  $-50\%$  and  $+100\%$  of those in the midpoint standard for the most recent initial calibration.

## 10.2 Sample Screening:

10.2.1 All samples (waters and soils) are screened by GC/FID before preparation for loading onto the GC/MS purge and trap system. The screening procedure prevents contamination from high-level samples from being introduced into the instrumentation. Dilution

levels are determined from the sample screening, allowing for a more rapid concentration estimation and limiting reruns for dilutions.

10.2.2 Water samples: Approximately 1 mL of the sample is removed from the sample vial with a disposable pipet and placed into a 40-mL glass vial/PTFE-lined open-top screw cap. The cap is finger tightened with the PTFE-lined screw cap facing toward the sample. The vial is shaken for about 30 seconds. The sample from which 1 mL has been removed must be analyzed within 24 hrs.

10.2.3 Soil samples: The third sample vial is used directly for headspace screening.

10.2.4 1 mL of headspace gas is removed from the headspace of the sample vial (waters and soils) with a gas-tight syringe and injected directly into the injection port of the GC/FID. The headspace response is recorded on an integrator.

10.2.5 The headspace FID response is compared to the headspace FID response of a 100 ug/L water standard or 100 ug/kg soil sample prepared in 1 mL of reagent water or 1 g of standard soil.

10.2.6 Sample dilutions:

Samples requiring dilution based on the headspace screening response will be diluted as follows:

10.2.6.1 Appropriate aliquots of the water samples will be added to reagent water to a total volume of 10 mL.

10.2.6.2 Appropriate aliquots of the methanol from the vial containing 5 grams of soil sample in 10 mL of methanol will be added to 10 mL reagent water.

### 10.3 Preparation of Water Samples for Purge and Trap

10.3.1 Remove the plunger from a 10 mL gas-tight syringe with an open/shut valve and close the valve.

10.3.2 Open the sample vial and pour the sample into the to the 10 mL mark on the syringe.

10.3.3 Reinsert the plunger into the syringe, turn it upright, bring the plunger end to the 10 mL mark.

10.3.4 With a 10 uL microsyringe, add 10 uL of the surrogate/internal standard spiking solution to the 10 mL sample in the syringe by inserting the microsyringe needle through the 10-mL syringe valve. Close the syringe valve. The concentration of the surrogates and internal standards will be 20 ug/L.

10.3.5 If the sample is a matrix spike, add 10 uL of the calibration standard spiking solution to the sample. The concentration of the spiked compounds will be 20 ug/L.

10.3.6 If the sample is a standard or laboratory control spike follow the above procedure using laboratory reagent water and the appropriate amount of the calibration standard or laboratory control sample spiking solutions.

10.3.8 Open the valve on the appropriate purging chamber on the autosampler apparatus, insert the sample syringe into the injection port, open the valve on the syringe, inject the water sample into the purging chamber, and close the chamber valve.

#### 10.4 Preparation of Soil Samples for Purge and Trap

10.4.1 Soil sample vials will not be opened by the analyst prior to analysis (with the exception of the vial designated for percent moisture determination).

10.4.2 With a 10 uL microsyringe, add 10 uL of the surrogate/internal standard spiking solution to the sample by inserting the microsyringe needle through the sample vial septum. The concentration of the surrogates and internal standards will be 20 ug/L in the water extract and 40 ug/L in the soil sample.

10.4.3 If the sample is a matrix spike, add 10 uL of the calibration standard spiking solution to the sample by inserting the microsyringe needle through the sample vial septum. The concentration of the spiked compounds will be 20 ug/L in the water extract and 40 ug/L in the soil sample

10.4.4 Soil samples are attached to the purge and trap retrofit apparatus for analysis.

#### 10.5 Concentrator Operating Conditions:

10.5.1 Adsorbent trap: Supelco K (10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001)

10.5.2 Delivery pressure: ~ 30 psi Helium

10.5.3 Valve temp: 180°C

10.5.4 Transfer line temp: 180°C

10.5.5 Purge temp set point: 40°C

10.5.6 Purge program:

10.5.6.1 Purge: 6 minutes

10.5.6.2 Dry purge: 2 minutes

10.5.6.3 Desorb preheat : 270°C

10.5.6.4 Desorb: 270°C/2 min

10.5.6.5 Bake : 275°C/4 min, bake gas bypass: 120 sec

10.5.6.6 Heater pockets preheat: 50°C/2 min

10.6 Autosampler Operating Conditions:

10.6.1 Valve temp: 175°C

10.6.2 Transfer line temp: 175°C

10.7 GC/MS Operating Conditions:

10.7.1 GC Operating Conditions For Full 8260 Compound List:

10.7.1.1 Column: DB624, 20m x 0.18m, 1.0mm film

10.7.1.2 Injector: 4mm ID low volume glass insert

10.7.1.3 Injector temp: 220°C

10.7.1.4 Detector temp: 280°C

10.7.1.5 Oven temperature program:

10.7.1.5.1 Initial temp: 35°C, hold for 4 minutes

10.7.1.5.2 Ramp temp: 8°C/min

10.7.1.5.3 Final temp: 200°C, hold for 0.5 minutes

10.7.1.6 Column flow : constant flow, 4.0 psi @ 35°C

10.7.1.7 Split flow: 20:1

10.7.2 MS Operating Conditions:

10.7.2.1 Mass range: 35-250 amu

10.7.2.2 Scan time: 2 sec/scan

10.7.2.3 Source Temp: 280 °C

10.8 Analytical Run Sequence:

10.8.1 BFB Tuning

10.8.2 Method blank

10.8.3 Initial calibration

10.8.3.1 1 ug/L calibration standard

10.8.3.2 5 ug/L calibration standard

10.8.3.3 10 ug/L calibration standard

10.8.3.4 20 ug/L calibration standard

10.8.3.5 50 ug/L calibration standard (use for daily calibration)

10.8.3.6 100 ug/L calibration standard

10.8.4 Method blank

10.8.5 Laboratory control sample (second source standard)

10.8.6 Method blank

10.8.7 Samples (including MS and MSD)

10.8.8 Method blank

10.8.9 Calibration verification standard (every 12 hours)

## 11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative analysis:

Qualitative identification of each target compound is based on retention time and comparison of the sample mass spectrum with the characteristic ions in the reference mass spectrum – the three ions of greatest intensity or any ions over 30% relative intensity. Compounds are identified as present in the sample when the following criteria are met:

11.1.1 The characteristic ions of a compounds is maximized in the same scan or within one scan of each other.

11.1.2 The retention time of the compound in the sample is with  $\pm 6$  seconds of the retention time of the compound in the calibration standard.

11.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.1.4 Structural isomers that produce similar mass spectra and are sufficiently resolved, should be identified as individual isomers.

## 11.2 Quantitative analysis

11.2.1 The quantitation of identified target analytes is based on the integrated abundance of the extracted ion current profile of the primary and secondary characteristic ion(s). These are listed in Table 5, p. 37-39, of Reference 14.1. The internal standard used for quantitation is the one nearest to the retention time of the analyte.

11.2.2 The average response factors from initial calibration are used to calculate the concentration of each compound in the sample using the following equation:

$$C_s = \frac{A_s \times C_{is} \times D}{A_{is} \times RRF \times V_s(M_s)}$$

$A_s$  = peak area of analyte in sample

$C_{is}$  = concentration of internal standard

$D$  = dilution factor

$A_{is}$  = peak area of internal standard

$RRF$  = mean response factor

$V_s (M_s)$  = volume of mass of sample

$$RRF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

$A_s$  = peak area of analyte

$A_{is}$  = peak area of internal standard

$C_s$  = concentration of analyte

$C_{is}$  = concentration of internal standard

## 12.0 DATA ASSESSMENT, QC CRITERIA, AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

12.1 Data is initially reviewed by the analyst for acceptability.

12.2 The table below lists the corrective actions that the analyst will follow if QC criteria are not initially met.

12.3 If the corrective action fail to correct the problem, the analyst must notify the client in the field and the KB Labs operations or QA officer for a decision on data usability.

**Table 10.3**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action(s)
Initial Demonstration of Capability (IDOC) – 4 replicate standard matrix spikes of all target analytes @ 10 - 50 times MDL	Prior to analysis of any samples	Historical lab acceptance limits, but not to exceed 70 – 130 percent.	Prepare and reanalyze new samples. Recalibrate, if necessary.
GC/MS Tuning – 4-bromofluorobenzene (BFB)	Prior to initial calibration and every 12 hours of analysis time	BFB ion abundance criteria must be met as listed in method.	Retune instrument. If necessary, clean source.
Initial Calibration (5 concentration levels) for all target analytes. Lowest conc. level at reporting limit.	Prior to sample analysis	The RSD of target analyte RFs must be $\leq$ 15%. Minimum mean RFs of SPCCs as listed in method must be met during initial calibration. The RSD of CCC RFs during initial calibration must be $\leq$ 30%.	Rerun calibration standards. Clean purge and trap transfer lines. Rerun initial calibration. Check for system leaks, clip six inches off column, change column. If necessary, prepare new calibration standards.
Calibration Verification (– a midlevel standard run every 12 hrs) prepared from	Daily before sample analysis and every 12 hrs of analysis time.	RF criteria for SPCCs the same as during initial calibration. RF of CCCs must be $\leq$ 20 percent difference from	Rerun CCS. Then rerun initial calibration, if necessary.

separate source from calibration standards		initial calibration. The IS retention times must be < 30 secs from those in the midpoint standard of most recent initial calibration. The IS responses must be within -50% to +100% of those in the midpoint standard of the most recent initial calibration.	
Method Blank	One per daily analysis batch.	No target analyte detected $\geq$ 5% or MRL	Bake out purge and trap system. Change adsorbent trap. Re- prep and reanalyzed method blank with associated samples.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) – all target analytes spiked at same conc. as LCS	One MS/MSD every 20 samples per matrix.	Should be within control limits established by lab.	Check LCS to determine if matrix effects apply.
Laboratory Control Sample (LCS) – all target analytes spiked at $\leq$ 50% of linear range calibrated. Prepared same as CVS.	One per daily analysis batch.	Must be within control limits established by lab.	Reprep and reanalyze LCS. Reanalyzed associated samples.
Surrogates – 4-Bromofluorobenzene, 1,2-Dichloroethane-d4, Toluene-d8, 1,4-Dichlorobenzene.	All samples, spikes, standards, and method blanks.	Must be within control limits established by lab or the method.	Reanalyze sample. If one or more still remain outside criteria, recalibrate and or remake surrogate solution.
Internal Standards - Fluorobenzene, Chlorobenzene-d5, 1,4-dichlorobenzene-d4.	All samples, spikes, standards, and method blanks.	Area must be -50 to +100% of last calibration check. RT must be $\pm$ 30 secs from last calibration check.	Reanalyze sample. If one or more still remain outside criteria, recalibrate and or remake IS solution.

### **13.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA**

- 13.1 Even though data that is out-of-control might be considered unusable, its usability will be decided after review and discussion between the client and KB Labs.
- 13.2 Out-of-control or unacceptable data will be provided with a definite qualifier and an explanation in the project narrative on the final report to the client.

### **14.0 METHOD PERFORMANCE**

14.1 Method performance is established by determining the Method Detection Limits (MDLs) in the matrix of interest. The 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 MDL that is achieved for a given analyte will vary depending on instrument sensitivity and matrix effects.

14.2 The MDL for both waters and soils is experimentally determined using procedures described in 40 CFR, Part 136, Appendix B and as per 91-04.

14.2.1 Seven replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest.

14.2.2 The concentrations for each analyte are then experimentally determined using the procedures described above for this method.

14.2.3 The standard deviation of the found concentrations for the seven replicates is then calculated.

14.2.4 The MDL for each analyte is then determined by multiplying the standard deviation by 3.

### **15.0 WASTE MANAGEMENT AND POLLUTION PREVENTION**

Refer to procedures described in KB Labs' *Standard Operating Procedure SOP010 (Waste Disposal)* and KB Labs' *Health and Safety Manual*.

### **16.0 REFERENCES**

14.1 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846, Method 8260C*, Revision 2, December 1996

14.2 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846, Method 8000C, Revision 2, December 1996*

14.3 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846, Method 5030B, Revision 2, December 1996*

14.4 *Test Method for Evaluating Solid Wastes, Fourth Edition, SW846, Method 5035A, Revision 0, December 1996*

14.5 Code of Federal Regulations, Title 40, Part 40, Appendix B.

14.6 Hewlett-Packard 59721 MSD Hardware Manual Number.

14.7 Tekmar LSC 2000 Purge and Trap Concentrator User Manual.

14.8 KB Labs' Quality Assurance Manual, August, 2003.

14.9 KB Labs' Health and Safety Manual, 1998.

14.10 KB Labs Standard Operating Procedure (SOP) No. 007, *Sample Receipt and Acceptance*, July 2003, Revision 1.

# ATTACHMENTS

METHOD DETECTION LIMIT STUDY													
Method: 8260B		Instrument I.D.: HP5890GC/HP5971A MSD											
Sample Prep Method: 5030B		Analytical Column: DB624, 20 m x 0.18 mm I.D., 1.0 um film thickness											
Analyst: Brad Weichert		Matrix: Water											
Date: 7/08/04		Sample Volume: 10 mL											
		Spike Conc. and Amount: 5.0 uL of 2 ug/mL spiking solution into 10 mL water											
Contaminant	Test Conc. (ug/L)	MDL Replicates (ug/L)										MDL	MRL
		1	2	3	4	5	6	7	X	SD			
Dichlorodifluoromethane	1	0.86	0.67	0.72	0.64	0.84	0.68	0.86	0.75	0.10	0.3	1	
Chloromethane	1	0.82	0.78	0.70	0.77	0.86	0.84	0.99	0.82	0.09	0.3	1	
Vinyl Chloride	1	0.81	0.65	0.75	0.84	0.84	0.74	0.76	0.77	0.07	0.2	1	
Bromomethane	1	0.71	0.70	0.83	0.88	1.13	0.67	0.81	0.82	0.16	0.5	1	
Chloroethane	1	1.06	0.78	0.75	0.98	0.97	0.70	0.77	0.86	0.14	0.4	1	
Trichlorofluoromethane	1	0.85	0.78	0.81	0.71	0.89	0.76	1.06	0.84	0.11	0.4	1	
1,1-dichloroethene	1	0.98	0.69	0.70	0.78	0.95	0.78	0.68	0.79	0.12	0.4	1	
Methylene Chloride	1	1.03	0.60	0.92	1.19	1.17	1.08	0.93	0.99	0.20	0.6	1	
t-1,2-Dichloroethene	1	0.98	0.62	0.74	0.96	0.97	0.81	0.87	0.85	0.14	0.4	1	
MtBE	1	0.73	0.66	0.97	0.87	1.04	0.90	0.99	0.88	0.14	0.4	1	
1,1-Dichloroethane	1	0.87	0.69	0.87	0.83	0.98	0.86	0.82	0.85	0.09	0.3	1	
2,2-Dichloropropane	1	1.01	0.79	0.87	0.90	0.80	0.85	0.78	0.86	0.08	0.3	1	
c-1,2-Dichloroethene	1	0.85	0.67	0.81	0.84	1.03	0.92	0.77	0.84	0.11	0.4	1	
Chloroform	1	0.78	0.60	0.89	1.00	0.99	1.02	0.95	0.89	0.15	0.5	1	
1,1,1-Trichloroethane	1	0.87	0.70	0.66	0.82	0.90	0.86	0.72	0.79	0.10	0.3	1	
Carbon tetrachloride	1	2.47	2.27	2.29	2.53	2.42	2.47	2.43	2.41	0.10	0.3	1	
Benzene	1	0.79	0.64	0.81	0.76	0.84	0.87	0.84	0.79	0.08	0.2	1	
1,2-Dichloroethane	1	1.11	0.75	0.92	1.07	1.17	0.85	1.11	1.00	0.16	0.5	1	
Trichloroethene	1	0.78	0.69	0.79	0.87	0.88	0.82	0.73	0.79	0.07	0.2	1	
1,2-Dichloropropane	1	0.80	0.71	0.74	0.88	0.99	0.88	0.74	0.82	0.10	0.3	1	
Dibromomethane	1	1.22	0.80	0.91	1.03	1.29	1.12	1.01	1.05	0.17	0.5	1	
Bromodichloromethane	1	0.74	0.62	0.91	0.74	1.02	0.82	0.97	0.83	0.14	0.4	1	
c-1,3-Dichloropropene	1	0.81	0.69	0.74	0.89	0.95	0.83	0.83	0.82	0.09	0.3	1	
Toluene	1	0.82	0.58	0.68	0.67	0.70	0.70	0.72	0.70	0.07	0.2	1	
t-1,3-Dichloropropene	1	0.79	0.86	0.70	0.81	0.93	0.98	0.83	0.84	0.09	0.3	1	
1,1,2-Trichloroethane	1	0.89	0.75	1.12	0.93	1.09	0.85	0.93	0.94	0.13	0.4	1	
Tetrachloroethene	1	0.84	0.74	0.73	0.73	0.94	0.96	0.84	0.83	0.10	0.3	1	
1,3-Dichloropropane	1	0.77	0.72	0.73	0.75	0.92	0.79	0.85	0.79	0.07	0.2	1	
Dibromochloromethane	1	0.90	0.58	0.53	0.77	0.74	0.92	0.77	0.74	0.15	0.5	1	
1,2 Dibromomethane	1	0.92	0.72	0.85	0.81	0.96	0.97	0.86	0.87	0.09	0.3	1	
Chlorobenzene	1	0.80	0.68	0.72	0.76	0.90	0.83	0.76	0.78	0.07	0.2	1	
1,1,1,2-Tetrachloroethane	1	0.89	0.64	0.68	0.75	0.77	0.84	0.76	0.76	0.09	0.3	1	
Ethylbenzene	1	0.73	0.66	0.66	0.68	0.74	0.75	0.75	0.71	0.04	0.1	1	
m&p-Xylene	1	1.42	1.51	1.25	1.45	1.44	1.43	1.43	1.42	0.08	0.3	1	
o-Xylene	1	0.82	0.83	0.62	0.70	0.75	0.71	0.81	0.75	0.08	0.2	1	
Styrene	1	0.69	0.71	0.74	0.66	0.93	0.78	0.86	0.77	0.10	0.3	1	
Bromoform	1	0.61	0.60	1.14	0.71	1.09	0.90	0.83	0.84	0.22	0.7	1	
Isopropylbenzene	1	0.76	0.75	0.67	0.68	0.73	0.78	0.72	0.73	0.04	0.1	1	
Bromobenzene	1	0.98	0.69	0.60	0.80	0.81	0.75	0.73	0.77	0.12	0.4	1	
1,1,2,2-Tetrachloroethane	1	0.94	0.85	0.73	0.81	0.95	0.69	0.95	0.85	0.11	0.3	1	
n-Propylbenzene	1	0.76	0.77	0.64	0.69	0.75	0.84	0.73	0.74	0.06	0.2	1	
2-Chlorotoluene	1	0.79	0.81	0.72	0.79	0.84	0.71	0.78	0.78	0.05	0.1	1	
4-Chlorotoluene	1	0.87	0.71	0.67	0.70	0.89	0.81	0.75	0.77	0.09	0.3	1	

1,3,5-Trimethylbenzene	1	0.68	0.68	0.68	0.68	0.75	0.76	0.82	0.72	0.06	0.2	1
tert-Butylbenzene	1	0.74	0.75	0.64	0.74	0.72	0.76	0.77	0.73	0.04	0.1	1
1,2,4-Trimethylbenzene	1	0.80	0.70	0.59	0.71	0.77	0.68	0.73	0.71	0.07	0.2	1
sec-Butylbenzene	1	0.74	0.71	0.64	0.70	0.75	0.86	0.75	0.74	0.07	0.2	1
1,3-dichlorobenzene	1	0.88	0.71	0.69	0.75	0.87	0.68	0.75	0.76	0.08	0.3	1
p-Isopropyltoluene	1	0.83	0.72	0.57	0.66	0.75	0.73	0.70	0.71	0.08	0.3	1
1,4-dichlorobenzene	1	0.77	0.90	0.81	0.77	0.82	0.91	0.87	0.84	0.06	0.2	1
1,2-Dichlorobenzene	1	0.82	0.80	0.65	0.84	0.82	0.86	0.87	0.81	0.07	0.2	1
n-Butylbenzene	1	0.77	0.78	0.65	0.68	0.83	0.84	0.78	0.76	0.07	0.2	1
1,2-Dibromo-3-chloropropan	1	0.64	1.86	1.69	1.63	2.17	2.81	1.12	1.70	0.70	2.2	5
1,2,4-Trichlorobenzene	1	0.77	0.66	0.77	0.96	1.07	1.18	0.90	0.90	0.18	0.6	1
Hexachlorobutadiene	1	0.84	0.79	0.53	0.77	0.91	1.04	0.99	0.84	0.17	0.5	1
Naphthalene	1	0.89	0.88	0.76	0.80	0.97	1.72	1.07	1.01	0.33	1.0	5
1,2,3-Trichlorobenzene	1	0.76	0.61	0.75	0.83	1.06	1.17	0.91	0.87	0.19	0.6	1
1,4-Dioxane	100	100	85	100	120	101	137	104	107	17	53	100

METHOD DETECTION LIMIT STUDY													
Method: 8260B		Instrument I.D.: HP5890GC/HP5971A MSD											
Sample Prep Method: 5035B		Analytical Column: DB624, 20 m x 0.18 mm I.D., 1.0 um film thickness											
Analyst: Brad Weichert		Matrix: Soil											
Date: 7/08/04		Sample Mass: 5 g											
		Spike Conc. and Amount: 5.0 uL of 2 ug/mL spiking solution into 5.0 g soil											
Contaminant	Test Conc. (ug/kg)	MDL Replicates (ug/kg)										MDL	MRL
		1	2	3	4	5	6	7	X	SD			
Dichlorodifluoromethane	2	1.96	2.04	2.10	1.86	1.58	2.04	1.88	1.92	0.17	0.5	2	
Chloromethane	2	1.70	1.62	2.42	1.56	1.96	2.04	2.12	1.92	0.31	1.0	2	
Vinyl Chloride	2	1.78	1.54	2.12	1.72	1.80	1.96	1.90	1.83	0.19	0.6	2	
Bromomethane	2	1.90	2.12	3.08	2.24	2.16	1.70	1.94	2.16	0.44	1.4	2	
Chloroethane	2	1.80	1.80	1.82	1.88	1.84	1.90	1.96	1.86	0.06	0.2	2	
Trichlorofluoromethane	2	2.12	2.00	2.06	1.78	1.68	1.78	1.78	1.89	0.17	0.5	2	
1,1-dichloroethene	2	2.04	2.20	2.34	2.04	1.70	2.22	1.86	2.06	0.22	0.7	2	
Methylene Chloride	2	2.20	2.38	2.48	1.68	1.60	1.76	1.88	2.00	0.35	1.1	2	
t-1,2-Dichloroethene	2	1.90	1.54	2.50	2.20	1.58	1.94	2.04	1.96	0.34	1.1	2	
MtBE	2	2.42	2.24	2.76	2.10	2.08	2.00	1.96	2.22	0.28	0.9	2	
1,1-Dichloroethane	2	1.72	1.72	2.18	1.56	1.72	1.96	2.02	1.84	0.22	0.7	2	
2,2-Dichloropropane	2	2.14	2.20	2.64	1.72	1.44	2.00	1.94	2.01	0.38	1.2	2	
c-1,2-Dichloroethene	2	1.50	1.70	1.96	1.78	1.92	1.90	1.76	1.79	0.16	0.5	2	
Chloroform	2	2.14	1.88	2.12	1.96	2.04	2.30	1.94	2.05	0.14	0.5	2	
1,1,1-Trichloroethane	2	1.60	1.82	2.04	1.54	1.80	1.76	1.88	1.78	0.17	0.5	2	
Carbon tetrachloride	2	4.58	4.94	4.52	4.66	4.58	4.56	4.94	4.68	0.18	0.6	2	
Benzene	2	1.86	1.82	1.96	1.54	1.70	1.86	1.92	1.81	0.14	0.5	2	
1,2-Dichloroethane	2	2.82	2.30	2.78	1.84	1.80	2.34	2.36	2.32	0.40	1.3	2	
Trichloroethene	2	1.94	1.76	1.82	1.52	1.80	1.96	1.92	1.82	0.15	0.5	2	
1,2-Dichloropropane	2	2.28	1.98	1.90	1.94	2.04	2.02	1.82	2.00	0.15	0.5	2	
Dibromomethane	2	2.44	2.06	2.98	1.84	1.86	1.68	2.38	2.18	0.45	1.4	2	
Bromodichloromethane	2	2.12	2.00	2.24	1.80	1.90	2.06	2.04	2.02	0.14	0.5	2	
c-1,3-Dichloropropene	2	1.76	1.86	2.08	1.60	2.02	2.10	1.94	1.91	0.18	0.6	2	
Toluene	2	1.72	1.70	1.82	1.56	1.62	1.58	1.94	1.71	0.14	0.4	2	
t-1,3-Dichloropropene	2	1.82	1.62	2.38	1.60	1.68	1.86	1.78	1.82	0.27	0.8	2	
1,1,2-Trichloroethane	2	2.32	1.82	2.10	2.06	1.58	1.72	1.82	1.92	0.25	0.8	2	
Tetrachloroethene	2	2.04	1.68	1.96	1.60	1.94	2.28	1.98	1.93	0.23	0.7	2	
1,3-Dichloropropane	2	2.48	1.96	2.18	1.82	1.94	2.26	1.82	2.07	0.25	0.8	2	
Dibromochloromethane	2	1.74	1.90	2.12	1.20	1.62	1.92	1.54	1.72	0.30	0.9	2	
1,2 Dibromomethane	2	2.80	1.86	2.32	1.36	1.72	1.86	2.08	2.00	0.46	1.4	2	
Chlorobenzene	2	1.92	1.80	1.78	1.56	1.70	1.94	1.84	1.79	0.13	0.4	2	
1,1,1,2-Tetrachloroethane	2	1.62	1.50	2.18	1.50	1.88	1.70	1.50	1.70	0.25	0.8	2	
Ethylbenzene	2	1.90	1.78	1.90	1.56	1.54	1.80	1.88	1.77	0.15	0.5	2	
m&p-Xylene	2	3.46	3.08	3.38	3.02	3.42	3.00	3.28	3.23	0.20	0.6	2	
o-Xylene	2	1.84	1.68	2.16	1.78	1.70	1.62	1.88	1.81	0.18	0.6	2	
Styrene	2	1.72	1.56	1.94	1.42	1.56	1.66	1.76	1.66	0.17	0.5	2	
Bromoform	2	1.76	2.16	2.22	1.62	2.68	1.86	1.68	2.00	0.38	1.2	2	
Isopropylbenzene	2	1.88	1.60	1.64	1.42	1.50	1.82	1.76	1.66	0.17	0.5	2	
Bromobenzene	2	2.26	1.64	1.54	1.56	1.74	2.14	2.00	1.84	0.29	0.9	2	
1,1,2,2-Tetrachloroethane	2	2.44	1.98	2.22	1.40	1.78	2.02	2.18	2.00	0.34	1.1	2	
n-Propylbenzene	2	1.84	1.68	1.66	1.58	1.58	1.80	1.88	1.72	0.12	0.4	2	
2-Chlorotoluene	2	1.74	1.74	2.18	1.60	1.60	1.80	1.88	1.79	0.20	0.6	2	
4-Chlorotoluene	2	2.14	1.64	2.02	1.64	1.60	1.90	1.94	1.84	0.21	0.7	2	
1,3,5-Trimethylbenzene	2	1.98	1.58	1.64	1.58	1.48	1.84	1.78	1.70	0.18	0.6	2	

tert-Butylbenzene	2	1.84	1.62	1.76	1.56	1.40	1.60	1.66	1.63	0.14	0.4	2
1,2,4-Trimethylbenzene	2	1.84	1.60	1.66	1.44	1.64	1.72	1.80	1.67	0.13	0.4	2
sec-Butylbenzene	2	1.76	1.72	1.76	1.40	1.46	1.72	1.78	1.66	0.16	0.5	2
1,3-dichlorobenzene	2	2.20	1.36	1.60	1.66	1.66	1.76	1.92	1.74	0.26	0.8	2
p-Isopropyltoluene	2	2.10	1.58	1.72	1.50	1.78	1.62	1.88	1.74	0.20	0.6	2
1,4-dichlorobenzene	2	2.36	1.58	1.98	1.64	1.98	2.06	2.20	1.97	0.28	0.9	2
1,2-Dichlorobenzene	2	2.26	1.74	2.26	1.72	1.58	1.86	2.02	1.92	0.27	0.8	2
n-Butylbenzene	2	1.94	1.84	1.84	1.54	1.54	1.76	2.12	1.80	0.21	0.7	2
1,2-Dibromo-3-chloropropan	2	2.94	3.66	2.70	2.10	3.90	4.22	4.08	3.37	0.80	2.5	5
1,2,4-Trichlorobenzene	2	4.58	2.22	2.82	2.00	2.00	3.04	2.60	2.75	0.90	2.8	5
Hexachlorobutadiene	2	3.14	1.44	2.54	1.70	2.02	2.24	2.16	2.18	0.56	1.7	2
Naphthalene	2	6.38	2.60	4.40	1.94	2.20	2.46	2.92	3.27	1.59	5.0	5
1,2,3-Trichlorobenzene	2	3.14	1.44	2.54	1.70	2.02	2.24	2.16	2.18	0.56	1.7	5
1,4-Dioxane	200	174	102	200	108	134	158	118	142	37	115	200

# Laboratory Quality Manual

Prepared by:

KB Labs, Inc.  
6821 SW Archer Road  
Gainesville, Florida 32608  
(352) 367-0073

In Accordance with:

Chapter 64E-1 Florida Administrative Code (FAC)  
Certification of Environmental Testing Laboratories  
And with the consensus standards adopted at the National Environmental  
Laboratory Accreditation Conference (NELAC)

This manual covers the following mobile units of KB Labs, Inc:

KB-1, KB-2, and KB-3

Effective Date: September 2008

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KB-1, KB-2, and KB-3

CONCURRENCES:

KB Labs, Inc. Laboratory Director:

Signature: \_\_\_\_\_  
Bradley A. Weichert

Date: \_\_\_\_\_

KB Labs, Inc. Quality Assurance Officer:

Signature: \_\_\_\_\_  
Michael G. Winslow

Date: \_\_\_\_\_

EFFECTIVE DATE: September 2008

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## **1.0 STATEMENT OF POLICY AND OBJECTIVES**

The policy of the management of KB Labs, Inc., is to implement a quality assurance program which is in compliance with the provisions and standards set forth in Chapter 64E-1 Florida Administrative Code (FAC), Certification of Environmental Testing Laboratories, which have been determined to be equivalent to the National Environmental Laboratory Accreditation Conference (NELAC) standards; and to assure that all certified environmental analyses are performed in accordance with the provisions and standards in Chapter 64E-1(FAC). The purpose is to ensure that all environmental data generated by KB Labs are scientifically valid, definable, and of known and acceptable precision and accuracy.

The management of KB Labs is committed to providing its clients services that conform to established quality requirements, including those associated with schedules and budgets, and assuring that all personnel strive to perform their job functions correctly without compromise of quality or obligations to clients.

### **1.1 SCOPE OF SERVICES**

KB Labs provides a number of on-site analytical services using mobile laboratory facilities. KB Labs performs chemical analyses only. The primary focus is the determination of

- Volatile organic compounds (VOCs) by gas chromatography/mass spectrometry (GC/MS), providing full confirmation data on-site.

## 2.0 STAFF ORGANIZATION AND RESPONSIBILITIES

Figure 2-1 shows the organization and line of authority of KB Labs personnel.

### 2.1 DESCRIPTION OF JOB RESPONSIBILITIES

The job descriptions of key staff are described below.

- **President** - responsible for all contractual obligations of the proposed work and directs corporate efforts as necessary to achieve the objectives of schedule, cost, and technical performance. The President is also responsible for the review and administration of all contract changes, and for the direct communication and liaison with the client. The President can also act as a project manager.
- **Director of Operations** - responsibilities include the preparation of work plans and schedules, the allocation of manpower and material resources, and the direct communication with field team operations. The Director of Operations can also act as project manager.
- **Quality Assurance (QA) Officer** - provides monitoring and periodic internal auditing of the quality control (QC) procedures of the field chemists, ensures that established QC procedures are being followed, that adequate documentation is provided, and that all QC problems are handled in an expeditious manner. The QA officer is also responsible for the formatting and quality control of all documents and for the compiling, updating and submitting of the forms, SOPs, and the Laboratory Quality Manual.
- **Laboratory (Technical) Director** - responsible for the overall technical operations of all mobile laboratory units. The Laboratory Director is responsible for certifying that the field chemists with the necessary educational and technical training perform the analytical tests and maintain the overall operation of each mobile unit in accordance with the policies and procedures documented in the Laboratory Quality Manual.
- **Field Chemists** - responsible for performing quality analytical work in accordance with published standard procedures. Field chemists serve primarily as chemical analysts, but may also function as project managers, field team leaders, sample custodians, couriers, or other capacities on a project-specific basis. Field chemists are generally assigned the responsibility of operating and maintaining a single field mobile unit.
- **Health and Safety Officer** - responsible for the oversight of the laboratory health and safety program and maintenance of the Health and Safety Manual.

## 2.2 PERSONNEL EXPERIENCE AND TRAINING

Documented evidence for the following will be maintained on an ongoing basis for each member of the organization in designated personnel files kept in the administrative office of KB Labs. Copies of these files will also be maintained in each mobile lab facility. This evidence will include resumes, training records, demonstrations of capability, results of performance evaluation samples, etc.

- All personnel shall have sufficient education, training, experience and technical knowledge to adequately meet the requirements and responsibilities of their designated functions in the organization and they must comply with the specific quality requirements of their function.
- Technical personnel must be able to demonstrate a specific knowledge and skill in the performance of their technical tasks, as well as a general knowledge of analytical methods, laboratory operations, QA/QC procedures, and records maintenance.
- All technical personnel must have read and understood the Laboratory Quality Manual.
- All technical personnel must have read and understood all SOPs that address functions for which they are responsible.
- Field chemists must demonstrate on an annual basis proficiency in the test methods for which they perform. This proficiency requirement can be met with successful performance of a demonstration of capability, a blind PE sample, or at least four consecutive laboratory control samples.

Refer to KB Labs' Standard Operating Procedure (SOP) No. 027, *New Analyst Training*.

## 2.3 ETHICS TRAINING

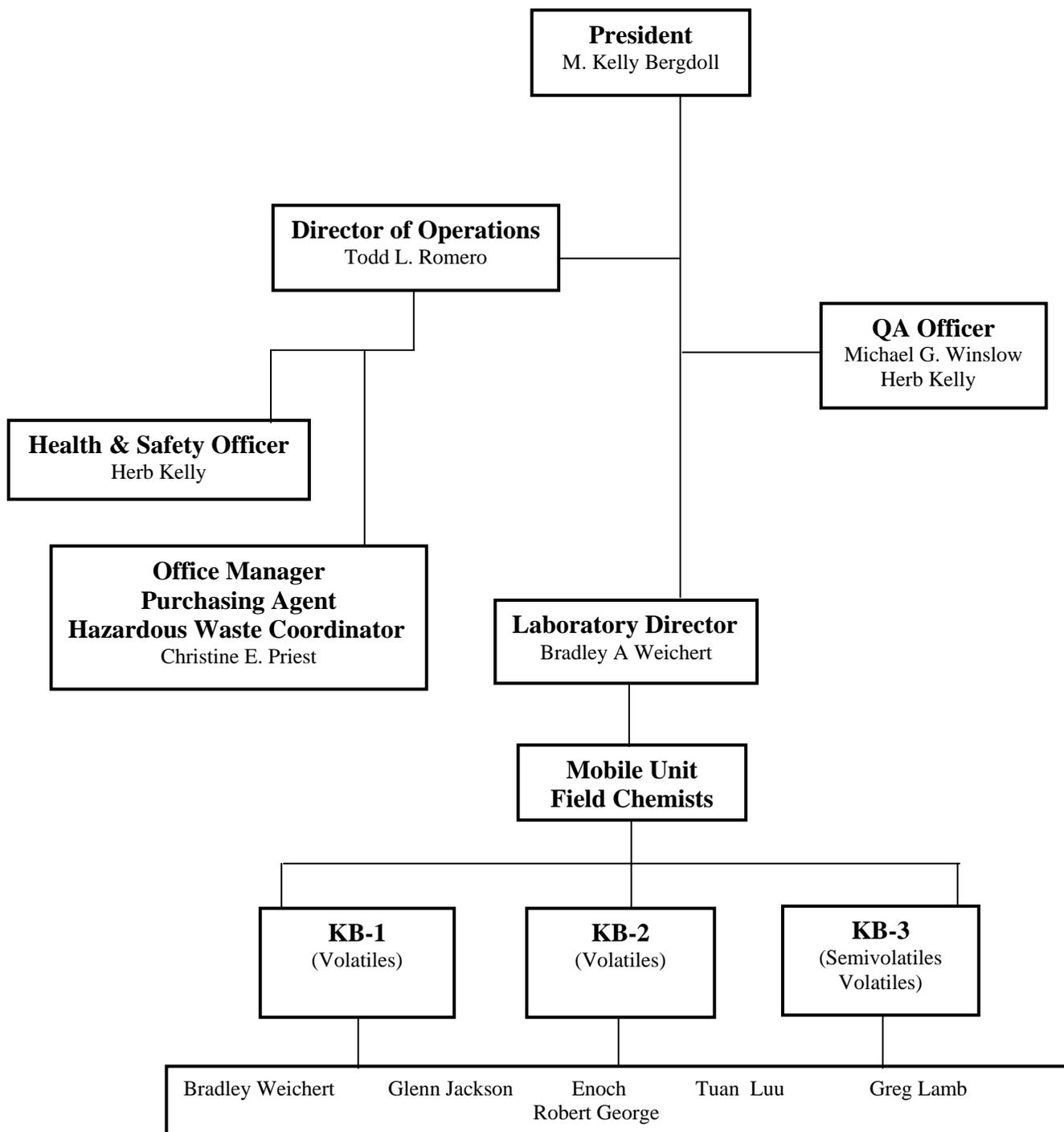
All employees will receive instruction in the basic standards of ethical conduct that are expected of them while employed by KB Labs. This training will be conducted at the beginning of their employment and on an annual basis thereafter. The training will be conducted by the Lab Director or the Quality Assurance Officer and will include matters relating to data falsification or manipulation, client confidentiality, and professional conduct.

- An employee who falsifies or improperly manipulates data will be subject to termination of employment and/or possible legal action.
- All data generated, collected, or obtained from a third party subcontractor by KB Labs about KB Labs clients will be treated as confidential. No information or analytical data will be provided to a third party without the permission of the client.

- Employees should use “common sense” in complying with acceptable business practices and at a minimum adhere to the following:
  - Accept or give no gifts that where it could be inferred that business favors might be returned or expected by KB Labs.
  - Do not use information gained as a KB Labs employee for personal gain.
  - Make no promises that conflict with the employee’s responsibilities to KB Labs.
  - Report any violations of company policies.
  - Comply with federal, state, and local laws and regulations governing personal and business conduct.
  
- Employees must read and understand KB Labs’ SOP No. 029, *Ethics and Individual Responsibility Training*. A signed and dated copy of this document will be placed in the employee’s training file.

#### 2.4 APPROVED SIGNATORIES

Table 2-1 below lists the approved title, current responsible party, and corresponding signature for laboratory document types.



**FIGURE 2-1: KB Labs Organization Chart**

**Table 2-1: Approved Signatories**

<u>Document Type</u>	<u>Title</u>	<u>Name</u>	<u>Signature</u>	<u>Initials</u>
Laboratory Quality Manual	QA Officer	Michael G. Winslow	_____	_____
	Lab Director	Bradley A. Weichert	_____	_____
Bid, Proposals	President	M. Kelly Bergdoll	_____	_____
	Director of Operations	Todd L. Romero	_____	_____
Contracts	President	M. Kelly Bergdoll	_____	_____
Reports to Clients	Director of Operations	Todd L. Romero	_____	_____
	President	M. Kelly Bergdoll	_____	_____
QA Reports	QA Officer	Michael G. Winslow	_____	_____
	QA Assistant	Herb Kelly	_____	_____
Preliminary Field Reports, Lab Notebooks, Logbooks, Data Sheets	Field Chemists	Bradley A. Weichert	_____	_____
		Greg G. Lamb	_____	_____
		Glenn Jackson	_____	_____
		Enoch	_____	_____
		Tuan Luu	_____	_____
Purchases	Office Manager	Robert George	_____	_____
		Christine Priest	_____	_____

### **3.0 FACILITIES AND EQUIPMENT**

KB Labs currently operates a total of three (3) mobile laboratories – two (2) dedicated to volatiles analysis, one (1) to semi-volatiles analysis and volatiles analysis. Each laboratory is designed to operate with a maximum of two field chemists, although generally for most projects only one chemist is required to operate a mobile laboratory facility. The mobile units are Izuzu single axle box trucks. Appendix A shows the floor plans for each of the mobile units with the arrangement of major analytical instrumentation and support equipment.

Tables 3-1 to 3-3 below list the major analytical instrumentation that is located in each of the mobile laboratories. Each mobile unit maintains the appropriate manuals supplied by the manufacturer for operation and maintenance of the instrumentation.

Each mobile unit operates as a stand alone laboratory and is NELAC certified as such.

**Table 3-1 Major Instrumentation, KB-1**

<u>Item(s)</u>	<u>Model(s)</u>	<u>Serial Nos.</u>	<u>Year Purchased</u>
Hewlett-Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 5890A / MSD 5971A*/ Chem Station	3235A46501 (GC) 3188A02953 (MSD)	1998 1998
Hewlett-Packard Gas Chromatograph/Flame Ionization Detector/Integrator	GC 5890A / FID 19231 / Integrator 3396	2643A09969 (GC)	1998
Tekmar Purge & Trap Concentrator / 16 Position Autosampler	LSC 2000/ ALS 2016	90288012 (LSC) 90277001 (ALS)	1998 1998

\* upgraded to 5972 in 2002

**Table 3-2 Major Instrumentation, KB-2**

<u>Item(s)</u>	<u>Model(s)</u>	<u>Serial Nos.</u>	<u>Year Purchased</u>
Hewlett – Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 6890A / MSD 5973A / Chem Station	US00041726 (GC) US92511963(MSD)	2006
Hewlett-Packard (HP) Gas Chromatograph/Flame Ionization Detector /Integrator	GC 5890A / FID 19231 / Integrator 3396	2541A06416 (GC)	1999
Tekmar Purge & Trap Concentrator / Varian Autosampler	LSC 3000 / Arcon	94271006 (Tekmar) 90178025 (Varian)	2006

**Table 3-3 Major Instrumentation, KB-3**

<b><u>Item(s)</u></b>	<b><u>Model(s)</u></b>	<b><u>Serial Nos.</u></b>	<b><u>Year Purchased</u></b>
Hewlett-Parkard (HP) Gas Chromatograph/Electron Capture & Flame Ionization Detectors/Integrator/Data System	GC 5890 / Integrator 3396 /Turbo-chrom 4.0	2750A1644470	1999
Hewlett – Packard (HP) Gas Chromatograph/Mass Spectrometer/Data System	GC 5890A / MSD 5971A / Chem Station	2643A09843 (GC) 3306A04459(MSD)	1999
Tekmar Purge & Trap Concentrator / Autosampler	LSC 2000 / ALS 2016	90248015 (LSC) 90178025 (ALS)	1999
Applied Separations Pressurized Solvent Extractor	PSE 10502	032000401	2001

## 4.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

Analytical reference methods and sample preparation reference methods currently performed by KB Labs, Inc. are listed in Table 4. The table also indicates the allocation of test methods among the different mobile units.

If additional, alternative, or modified procedures are ever proposed, a complete description of the method with data from an initial demonstration of proficiency will be provided to DOH for approval.

Each mobile unit maintains a copy of the most recent revision of the EPA SW846 reference method available for the tests performed in the mobile unit. In addition, a copy of the KB Labs analytical method SOP is attached to the published reference method. These SOPs document specific steps, procedural changes, and operating conditions actually utilized by KB Labs field chemists.

A comprehensive Laboratory Methods Manual is also maintained in the KB Labs administrative office that contains the latest revision of the SW846 methods used by KB Labs as well as copies of KB Labs analytical method SOPs.

### 4.1 ADDITIONAL SOPs

In addition to analytical method SOPs, KB Labs maintains copies of the following SOPs in both the mobile lab units and the administrative office:

SOP No.	Title
SOP001	Mobile Lab Power-Up
SOP002	Storage and Management of Gas Cylinders
SOP003	Final Report Preparation, Review, and Delivery
SOP004	Data Review and Validation
SOP005	Supply Requisition
SOP006	New Work Assignments
SOP007	Sample Receipt and Acceptance
SOP008	Filing and Archiving Project Records
SOP009	Significant Figures and Rounding Off
SOP010	Waste Disposal
SOP011	Temperature Monitoring

SOP No.	Title
SOP012	Storage of Standards
SOP013	Sample Storage
SOP014	Maintenance of Control Data
SOP015	Document Control
SOP016	Handling Complaints
SOP017	Corrective Actions
SOP018	Quality Control
SOP019	Labeling and Tracking of Standards
SOP020	Audits
SOP021	Sample Identification and Tracking
SOP022	Proficiency Test Samples
SOP023	Analytical Run Sequence
SOP024	Detection Limits
SOP025	Sample Containers, Preservation, and Holding Times
SOP026	Subsampling
SOP027	New Analyst Training
SOP028	Demonstration of Capability
SOP029	Ethics Training
SOP030	Contingency Plans for Changes in Ownership
SOP031	Protecting Confidentiality, Proprietary Rights, and National Security
SOP032	Departures from Documented Policies and Procedures
SOP033	Downtime Events
SOP034	Management Quality System Review
SOP035	Calculations
SOP036	Calculating and Reporting Soil Data
SOP037	Sample Receipt, Storage, and Disposal for Off-site or Fixed-base Analysis
SOP038	Subcontractor Pre-qualification Policy
SOP039	Weighing Soil Samples

SOP No.	Title
SOP040	Corrections to Entries
SOP041	Matrix Identification of Laboratory Control Samples
SOP042	Determining Measurement Uncertainty
SOP043	Manual Integration
SOP044	Calibration of Volumetric Dispensing Devices
SOP045	Limit of Detection

Table 4-1 Analytical Methods Performed by KB Labs, Inc.

<u>Parameter</u>	<u>Matrix</u>	<u>Sample Preparation Reference Method</u>	<u>Analytical Reference Method</u>	<u>KB Labs SOP No.</u>
Volatile organics (KB-1, KB-2, KB-3)	Water	5030B	8260B	KBSOP01VOC
	Soil	5035	8260B	

## 5.0 PROJECT OPERATIONS

Figure 5-1 shows a flowchart of KB Labs overall project operations and primary task responsibilities from field trip preparation to data report delivery.

### 5.1 PROCEDURES FOR NEW WORK ASSIGNMENTS

Before accepting a new work assignment, the Director of Operations reviews the Request for Proposal (RFP) and/ or verbally questions the potential client to ascertain the following elements to determine if KB Labs, Inc. can successfully complete the work assignment:

- Analytical compounds to be analyzed and by what analytical method.
- Determine sample matrix (i.e., ground water, soils, etc.)
- Approximate number of samples to be analyzed.
- Time duration and site location of project.
- Requested reporting limits.
- Point-of-Contact (POC) information
- Final reporting requirements (both electronic and hardcopy).

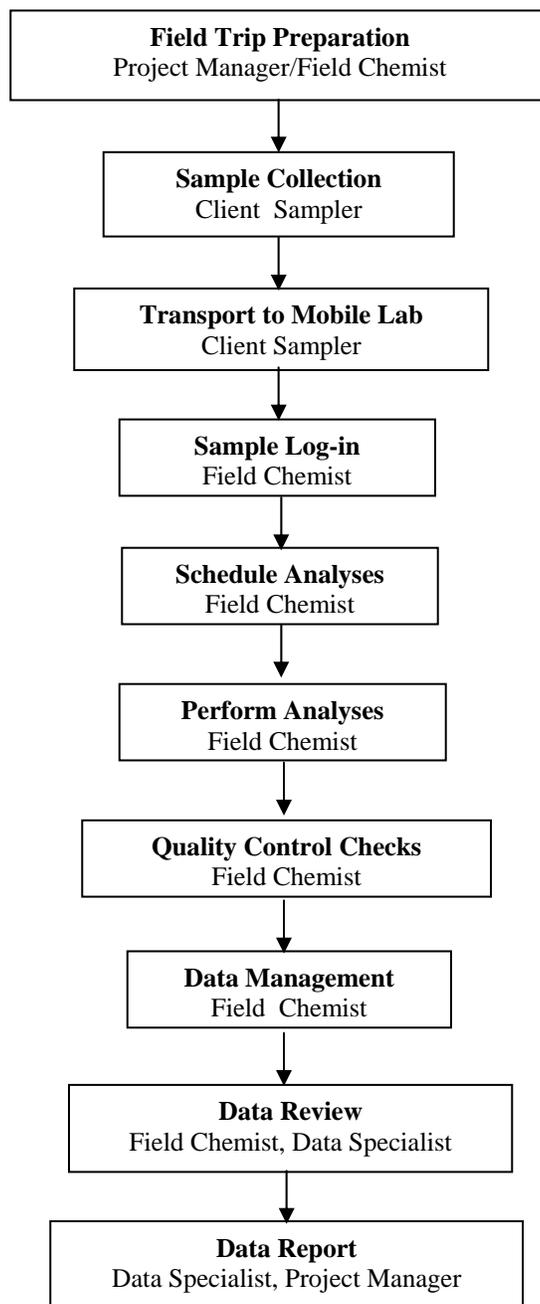
The following elements are reviewed by the Director of Operations as the basis for mobile laboratory and Field Chemist assignment to new project:

- Analytical compounds to be analyzed and analytical method.
- Training and analytical experience of Field Chemist.
- Availability of laboratory and Field Chemist for scheduled project dates.
- Past chemist experience at site and/or previous interaction with client.

Preparation of the laboratory and Field Chemist for new work assignment involves the following:

- Field Chemist is issued completed Work Order Form (See Appendix B) from the Director of Operations (Project Manager).
- Required standards, gases, and laboratory supplies are issued (if necessary) to the assigned laboratory by the Director of Operations.
- Routine maintenance is performed on the laboratory and analytical instruments prior to departure by the Field Chemist.

See also KB Labs' SOP No. 006, *New Work Assignments*.



**FIGURE 5-1: Flowchart of KB Labs Project Operations**

## 6.0 SAMPLE TRACKING AND HANDLING`

### 6.1 SAMPLING

KB Labs provides no sampling services. However, KB Labs will supply sample containers. All sample containers are purchased pre-cleaned (and with preservatives if required) from certified commercial suppliers. Sample containers are not cleaned for reuse.

Table 6-1 lists containers, preservation methods, and holding times for volatile and semi-volatile sample types.

### 6.2 SAMPLE CUSTODY

Sample custody is an essential part of field and laboratory operations and is defined as follows:

**Sample custody** – the sampler or transferee is in physical possession of the sample or was in physical possession of sample and sample was then placed in a secure area to prevent tampering. Where data may be needed for potential litigation, strict **chain-of-custody** procedures must be used.

### 6.3 CHAIN-OF-CUSTODY

A Chain-of-Custody Record (see Appendix B) is initiated at the time sample containers are dispatched to the field sampling team by the field chemist. A Chain-of-Custody Record accompanies sample containers to the field. This document is used by the field team to record sample identification, sample description, date, time, and location of collection, analyses required, and condition of sample. All requested information on this form must be completed where appropriate. This document must be signed by a member of the field team. All errors are deleted with one line through error and initialed and dated.

However, in order to meet the NELAC requirement that each sample have a **unique sample identification number** to facilitate and insure accurate sample tracking if needed at a later date, and because clients often repeat sample identification schemes from site to site, KB Labs will identify samples by combining the mobile lab identification, sampling date, and client field ID – e.g. for field sample SB-1, analyzed in mobile unit KB-1 and received from the client on January 1, 2006, its unique identification will be KB1\*010106\*SB-1. Refer to KB Labs SOP No. 021, *Unique Sample Identification*.

### 6.4 SAMPLE RECEIPT

The field chemist is designated as the sample custodian. This person receives samples from a member of the field sampling team and checks for the following:

- appropriate sample containers.

- adequate sample volume or mass
- signs of leaking, broken, or contaminated sample containers.
- whether headspace is present in the sample container (VOCs).
- proper preservation as specified in Table 6.
- complete documentation and identification of samples, and signature of field sampler on the Chain-of-Custody Record
- proper sample labeling

If any of the previous conditions are not properly met, the improper conditions for each sample will be noted on the Chain-of-Custody Record and will be reported to the field team leader. The field team leader will then make the decision on whether to reject the sample(s). After verifying the status of the samples, the sample custodian (field chemist) signs the sample Chain-of-Custody Record. The original copy is kept with the project file, a copy is placed in the Sample Receipt logbook, and another copy is sent to the client.

Refer to KB Labs' SOP No. 007, *Sample Receipt and Acceptance*.

## 6.5 SAMPLE STORAGE

All samples are stored in wet ice in coolers kept at  $\leq 6$  °C. Prior to sample preparation or analysis, samples are retrieved from the cooler by the field chemist and allowed to come to room temperature before analysis. The samples are returned to the cooler upon completion of sample preparation or analysis. (VOC samples are not returned to the cooler after aliquots are removed for processing. However, duplicate samples do remain in the cooler until disposal.)

The field chemist has the ultimate responsibility of ensuring analytical holding times are met for each project. All samples are secure in the laboratory with access only available to members of the staff of KB Labs and designated sample custodians. All samples are stored well away from standards. All samples for VOC analyses are stored on ice in a separate cooler at  $\leq 6$  °C. No other samples, reagents, extracts or standards will be stored in this cooler.

Refer to KB Labs' SOP No. 013, *Sample Storage*.

## 6.7 SAMPLE DISPOSAL

Unused samples should be returned in their containers to the client field samplers at the conclusion of the job, before the mobile lab leaves the project site. Purged and extracted samples and solvent extracts will be stored in clearly labeled approved containers and disposed of in accordance with DEP approved procedures.

Refer to KB Labs' SOP No. 013, *Waste Disposal*.

**Table 6-1: Sample Containers, Preservation Methods, and Holding Times\***

<u>Parameter</u>	<u>Matrix</u>	<u>Container</u>	<u>Preservative**</u>	<u>Holding *** Time (days)</u>
Volatiles	Water	Glass vial, screw cap with Teflon™-lined septum, 2 x 40 mL	Cool, ≤ 6 °C PH < 2	14
	Soil	Glass jar, Teflon™-lined screw cap, 4 oz.	Cool, ≤ 6 °C	14
Semivolatiles	Soil	Glass jar, Teflon™-lined screw cap, 4 oz.	Cool, ≤ 6 °C	14 extraction 40 analysis

\* From 40 CFR Part 136 Table II and Chapter 62-160 F.A.C.

\*\* Sample preservation should be performed immediately after sample collection.

\*\*\* Samples are analyzed on-site, generally within 24 hours of collection.

## 7.0 CALIBRATION AND TRACEABILITY OF MEASUREMENTS

Because measuring operations employing analytical instruments and other support test equipment have an effect on the accuracy or validity of tests, each mobile unit must perform calibration and verification procedures before equipment is put into service and on continuing basis.

### 7.1 INSTRUMENT CALIBRATION

Instrument calibration procedures establish the relationship between a calibration standard of known concentration and the measurement of the standard concentration by an instrument or analytical procedure. At a minimum, calibration is required (1) when an instrument is first started up; (2) daily, prior to the analysis of a batch of samples, (3) when the instrument has been subject to major maintenance, or (4) when the instrument fails the calibration quality control checks.

**Initial calibration** is performed when the instrument is started up or when the instrument response has drifted out of calibration in order to demonstrate that the instrument is capable of acceptable performance at the beginning of the analytical run and is producing a linear calibration. Initial calibration is usually performed with five standards that cover the analytical working range of the method. The standard concentrations will be adjusted to take into account the instrument and method, the upper and lower limits of linearity, and the instrumental detection limit.

**Continuing calibration** is performed at the beginning and/or every 12 hours in order to verify initial calibration. The continuing calibration standard (CCS) is generally a mid-level standard from the initial calibration but should be varied within the calibration range on a regular basis.

(Sample responses are quantitated from the initial calibration and not from continuing calibration.)

In all cases, if the method calibration requirements are more stringent than those listed in this document, then the method calibration requirements will be followed. In all cases, when an instrument is calibrated for analysis it will be recorded in an instrument logbook with date, initials of analyst, analyte(s), and all appropriate instrument settings. It will also be recorded on the analytical bench sheet or computer printout how the instrument was calibrated.

### 7.2 PREPARATION OF INSTRUMENT CALIBRATION STANDARDS

Stock solutions used to prepare calibration standards, surrogate and matrix spike solutions, and internal standard solutions are purchased from commercial suppliers (see Table 7-1 below).

For stock standards purchased directly through a supplier, the initials of the receiver and date of receipt are written in ink on the original Certificate of Analysis. If the standard has an expiration

date, this date is circled in ink to ensure that the preparer does not use expired standards. As these expire, they are disposed. All information concerning these standards, including LOT#, supplier of standard, concentration of standard, purity of standard, and method of determination of purity are on the original container and the Certificate of Analysis which accompanies the standard. Further information concerning the purchase of the standards is in a purchase order logbook with date of purchase, purchaser of standard, supplier of chemical and date of receipt of standard. All other information concerning these standards can be obtained from the supplier of the standard as needed. The original copy of the Certificate of Analysis for stock standards purchased is kept on file with the Quality Assurance Officer.

Working standards are prepared directly from the stock standard. If required, all dilutions are prepared in Class-A volumetric glassware. All documentation tracing the working standards to stock standards and chemicals will be kept in a standards notebook next to each instrument and will include the analyte(s), initials of analyst, date of preparation, concentration levels of standards, how standards were prepared, and stock standard used to prepare working standards and intermediate standards if applicable.

Working standard solutions will be prepared by sequential dilution of a single stock standard to bracket the analytical working range of the method. Working standard solutions may be either composite standards of more than one analyte or single-analyte solutions. The standard concentrations will be adjusted to take into account the instrument and method, upper and lower limits of linearity, and the instrumental detection limit. At least three (3) standard concentrations covering the working range and a blank will be prepared and analyzed. The working standards and the blank will be analyzed at the beginning of the analytical run (initial calibration) and at least one mid-level standard will be reanalyzed at least every 12 hours and at the end of the run to check for constant instrument response (continuing calibration verification).

### 7.3 STANDARD CURVE CALIBRATION

The working curve will be produced by plotting the standard response for each standard versus the concentration of each standard from the initial calibration run or average response factors, if method criteria are met. Specific quality control acceptance criteria for working curves or for continuing calibration standards are listed in the methods and will be followed.

### 7.4 INTERNAL STANDARDS (GC/MS)

Internal standards are added to all samples and standards that are analyzed for GC/MS analysis. Quantitation cannot be performed without internal standards. Method appropriate internal standards are listed in the analytical reference method. The working concentration of the internal standards will be prepared according to the method.

### 7.5 INSTRUMENT TUNING (GC/MS)

Daily instrument tuning will be performed to ensure that the instrument is calibrated and in proper working condition. Bromofluorobenzene (BFB) will be used as the tuning compound for volatile analysis and the mass intensity specifications will be followed according to each method. The working concentration for BFB will be prepared according to the method.

#### 7.6 STORAGE OF CALIBRATION AND REFERENCE STANDARDS

All standards are stored in refrigerators located in each mobile laboratory facility. Standards will be stored separately from samples. VOCs will be stored in a freezer at  $-10^{\circ}\text{C}$  and SVOCs and metals in a refrigerator at  $\leq 6^{\circ}\text{C}$ .

Refer to KB Labs' SOP No. 013, *Storage of Standards*.

#### 7.7 MONITORING OF REFRIGERATORS AND FREEZER

Temperatures for refrigerators and freezers are measured on a daily basis and recorded in a Daily Temperature Record . If the measured temperature for this equipment is out of control, it will be noted in the logbook, the necessary adjustment will be made to correct the temperature and it will be monitored until temperature is in control and constant. All laboratory thermometers are calibrated annually against a NIST certified thermometer. If any thermometer is more than  $1^{\circ}\text{C}$  different from the NIST thermometer, it is replaced.

**Table 7-1: Standard Sources and Preparation**

<u>Type</u>	<u>Standard Source</u>	<u>Preparation From Source</u>	<u>Lab Stock Storage</u>	<u>Preparation Frequency</u>
Calibration compounds	Purchased from supplier	Working solutions are made directly from source stock	Refrigerator @ $\leq 6\text{ }^{\circ}\text{C}$ (SVOCs) Freezer @ $-10\text{ }^{\circ}\text{C}$ (VOCs)	Weekly for gases, or monthly
GC/MS Internal standards (VOCs)	Purchased from supplier	Working solutions are made directly from source stock	Freezer @ $-10\text{ }^{\circ}\text{C}$	Semiannually
Matrix spike and surrogate compounds	Purchased from supplier	Working solutions are made directly from source stock	Refrigerator @ $\leq 6\text{ }^{\circ}\text{C}$ (SVOCs) Freezer @ $-10\text{ }^{\circ}\text{C}$ (VOCs)	Semiannually
GC/MS Tuning Compound Bromofluorobenzene	Purchased from supplier	Working solutions are made directly from source stock	Freezer @ $-10\text{ }^{\circ}\text{C}$	Annually

## 8.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA ACCURACY AND PRECISION

Data **accuracy** will be assessed for each measurement system and each sample lot using a known reference sample and/or a sample spiked at a known level. The recovery of the sample will then be compared to the method accuracy acceptance criteria established by the laboratory.

Data **precision** will be assessed similarly using replicate analyses. Data precision will be compared to the method precision acceptance criteria established by the laboratory.

If the accuracy or precision results do not fall within the established control limits for method performance, then the results reported for all samples processed as part of the same set must be labeled as suspect, and the samples may need to be repeated. The project QA officer and project manager will be notified and the necessary corrective action implemented.

In all cases, if the EPA method specific QC requirements (if established) are more stringent than those established by KB Labs, then the method QC requirements should be followed.

### 8.1 LABORATORY QUALITY CONTROL CHECKS

Types of QC samples used include method blanks, matrix spikes, matrix spike duplicates, laboratory control or reference spikes, surrogates, and blind performance evaluation samples.

The following minimum QC checks will apply to all analyses:

**Method blank** – Daily analysis of laboratory reagent water or standard soil samples is performed in order to monitor the cleanliness of the analytical system. Method blank analysis (VOCs only) is performed before analyzing samples, after high level sample analysis, and at least every 12 hours. For SVOCs there should be at least one method blank for every batch of 20 or less samples extracted.

**Matrix spike/matrix spike duplicates (MS/MSD)** – A known amount of each target compound is added to duplicate aliquots of a selected field samples in order to monitor the performance (precision and accuracy) of the target analytes in an actual matrix. An MS/MSD is analyzed at a frequency of one pair every 20 samples of a matrix type (soil or water).

**Reference standard/laboratory control spike (REF/LCS)** – A REF/LCS is analyzed after the initial calibration to check the validity of the calibration standards. The REF/LCS is prepared from a different source stock standard than are the calibration standards. An REF/LCS is analyzed at a frequency of one for every preparation batch of 20 or less samples of a matrix type.

**Surrogate standards** – The surrogate standard solution is added to all samples and standards that are analyzed. The surrogate compounds evaluate the performance of the analytical system and to help determine the potential for sample matrix effects.

QC tables are maintained for duplicate spikes, and reference samples. Separate tables are maintained for each analytical method. Warning and control limits are established by standard deviation techniques.

For duplicate samples, the relative standard difference (RPD) =  $\frac{|X_1 - X_2|}{(X_1 + X_2 / 2)} \times 100$

is utilized as the test statistic precision.

For spike data, the test statistic for accuracy is the percent recovery of the spike defined as follows:

$$\% \text{ Recovery} = \frac{\text{conc. spiked sample} - \text{conc. unspiked sample}}{\text{conc. of spike actually added}} \times 100$$

The test statistic for reference samples is the actual measured concentration. For both of these test statistics the mean and standard deviation are determined utilizing a number of data points. The warning and control limits are established as  $\pm 2$  and  $\pm 3$  standard deviation units from the calculated mean values, respectively. These limits are updated on a continuing basis as new QA data is entered. They are based on the most recent 50 data points for a given analyte and matrix. If a sufficient number of QA data points is not available for a given analyte and matrix, then the QA targets will be based upon published QA targets until sufficient data points have been generated.

Refer to KB Labs SOP No. 018, *Quality Control*.

## 8.2 METHOD PERFORMANCE

Method performance is established by determining the **Method Detection Limits (MDLs)** in the matrix of interest. The 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 MDL that is achieved for a given analyte will vary depending on instrument sensitivity and matrix effects.

The MDL for both waters and soils is experimentally determined by KB Labs using procedures described in 40 CFR, Part 136, Appendix B and as per 91-04. Seven replicate samples of each matrix (standard laboratory reagent water or soil) are spiked with a known concentration of each analyte of interest. The concentrations for each analyte are then experimentally determined using the procedures described above for this method. The standard deviation of the found concentrations for the seven replicates is then calculated. The MDL for each analyte is then determined by multiplying the standard deviation by 3.14.

Refer to KB Labs' SOP No. 024, *Detection Limits*.

**Laboratory control limits** are established by KB Labs for both waters and soils. The laboratory control limits are calculated by determining the average percent recovery and standard deviation measured for each analyte when determining its MDL. The upper and lower control limits are calculated as the average percent recovery plus or minus 3 times the standard deviation.

All MDLs will be verified or updated on an annual basis. Copies of the MDL studies for each method will be kept in the appropriate mobile units and in the mobile unit files maintained in the administrative office.

Refer to KB Labs' SOP No. 014, *Maintenance of Control Data*.

### 8.3 DEMONSTRATION OF ANALYTICAL CAPABILITY

Each analyst, prior to using any test method on samples, must perform a demonstration of capability for each method. This requirement will also hold for any time a new instrument is introduced into the laboratory.

The demonstration of capability will consist the same procedures followed for determining the MDLs as described in Section 8.2 above. A minimum of four (4) replicate samples (instead of 7) are required.

All demonstrations of capability will be documented on the Demonstration of Capability form shown in Appendix B. These completed and signed forms will be maintained in each analyst's training and experience file.

Refer to KB Labs' SOP No. 028, *Demonstration of Capability*.

## 9.0 DATA REDUCTION, VALIDATION, AND REPORTING

### 9.1 DATA REDUCTION

Data reduction and transfer are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and that accepted statistical techniques are used. Field chemists are responsible for calculating final data and QC data from raw data recorded on laboratory bench sheets, chart recordings, and computer printouts. All calculations are in accordance with the approved methods cited earlier. Example calculations are included with summarized data to facilitate review. All computer printouts should be labeled with analyst name, analyte, date of analysis, project name, and all pertinent instrument settings. All data are compiled in a project file folder for delivery to the Data Specialist for review. To facilitate data review for each project, the following items should be in each project file folder in the order listed:

- Diskette with field preliminary data report in electronic spreadsheet format
- Work Order Form
- Field log sheets
- Chain-of-custody sheets
- A hardcopy of the field preliminary data report
- Field Chemist Comments (See Appendix B) – any pertinent comments such as departures from method, problems, etc.
- Spike recovery summaries
- Initial multilevel calibration summaries (if performed)
- Tune records (MS)
- Daily sequence summaries
- Instrument analysis chromatograms and quantitation reports in chronological order
- Screening chromatograms

### 9.2 DATA REVIEW AND VALIDATION

Data review and validation is conducted by the Data Specialist who is responsible to the designated project manager. All work performed by the Field Chemist is checked during the data review process. The signature of the reviewer and date of review are entered on the Data Review Checklist (see Appendix B) each project file folder. The responsibility of the Data Specialist is to ensure the following:

- Each project folder has the items listed in Section 9.1 above.
- All data are calculated correctly.
- All data are entered correctly in the field data report.
- All QC data are calculated correctly.
- All QC values are within the acceptance criteria.
- Check that chain-of-custody forms are properly completed and that sample identifications are in agreement with those in the project file and data report.

Upon completion of data review and validation, the Data Specialist will include a Data Folder Table of Contents Checklist (see Appendix B and a Data Review Checklist in the project data file .

Refer to KB Labs' SOP No. 004, *Data Review and Validation*.

### 9.3 DATA REPORTING

The Data Specialist generates the final report to the client and is responsible to the designated project manager. Assurance that reported data are correct is the responsibility of the project manager, the Data Specialist, and the Field Chemist. The final report to the client contains the following:

- A cover letter which references the project name, date, and location and summarizes the contents of the report and the qualifications of KB Labs, Inc
- A brief project narrative which provides general information about the project scope, analytical procedures, analytical results, and QC data
- A data narrative addressing discrepancies between the final data report and the preliminary field data report
- A table listing the analytical run sequence with surrogate recoveries
- A table listing matrix spike and control spike recoveries
- A final data report in spreadsheet format
- Copies of the chain-of-custody sheets

Appendix C gives an example of a standard KB Labs final report to client.

Refer to KB Labs' No. 003, *Final Report Preparation, Review, and Delivery*.

## 10.0 SYSTEM AND PERFORMANCE AUDITS

Systems and performance audits are used to assess and document the performance of field chemists. These audits form a basis for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements. Refer to KB Labs' SOP No. 020, *Audits*.

### 10.1 INTERNAL SYSTEM AUDITS

The QA Officer will choose random sample numbers from different projects conducted during the year and trace the sample from receipt to final reporting, reviewing proper chain of custody, sample receipt procedures, proper method selection, data reduction, sample preparation, and analysis within holding times. At least one sample will be audited annual for each method and mobile unit. Completed Internal Audit Reports will be submitted to the Lab Director and to the President.

All laboratory QC information will be reviewed at least annually by the QA officer. This information, including replicates, spikes, and reference samples for all methods is printed and kept on file.

The QA officer checks all instrument and analytical logs to assure that all analytical work that has been done is properly documented, including date of analysis, initials of analyst, analyte, and all pertinent instrument settings.

### 10.2 EXTERNAL SYSTEM AUDITS

KB Labs is biannually inspected by FL DOH, and all recommendations are implemented. KB Labs is open to FL DOH for inspection at any time.

### 10.3 INTERNAL PERFORMANCE AUDITS

Internal performance audits consist of commercially produced QC check samples (Laboratory Control or Reference Samples) run with each analysis by the analyst. The found value is then compared to the true value. All QC check sample data is entered onto the analytical bench sheet including true value, found value, percentage recovery and, if applicable, 95 percent confidence interval. The QC check sample will be from a different stock source than the calibration standards.

#### 10.4 EXTERNAL PERFORMANCE AUDITS

Biannual performance audits include the analysis and evaluation of **proficiency test samples**. KB Labs will participate in the U.S. EPA Water Pollution Laboratory Performance Evaluation study Program. Results of these analyses will be provided to DOH.

Refer to KB Labs' SOP No. 022, *Proficiency Test Samples*.

## 11.0 CORRECTIVE ACTION

Data acceptability is based on the quality assurance objectives for measurement data stated in Section 8.0.

If the acceptance criteria are not met for one or more QC checks, than the first person to take corrective action will be the analyst. The analyst will determine when the system was no longer in control and follow the corrective action.

If the system is still out of control, the Laboratory Director and QA officer will be informed of the specific discrepancies and decisions concerning the data will be made on a project specific basis.

The Laboratory Director or QA officer will be responsible for notifying laboratory personnel of the corrective action(s) be taken.

For external QC discrepancies (performance evaluation sample), the Laboratory Director and QA officer will determine the source of the discrepancy, plan a course of action to solve the problem and inform the appropriate laboratory personnel of the new procedure.

All DOH recommended corrective actions will be initiated as a result of system or performance audits, split samples or data validation review.

Refer to KB Labs SOP No. 17, *Corrective Actions*.

### 11.1 COMPLAINTS

Whenever a complaint is received by KB Labs from a client or other party about compliance with the NELAC Standard, project requirements, laboratory policies and procedures, or about the quality of the laboratory's test results, an internal audit (Sec. 10.1) will be immediately conducted by the Quality Assurance Officer, or in the case of specific project issues unrelated to data quality, the Director of Operations will investigate the problem. A record of the complaint and subsequent action will be maintained in the project file in the administrative office.

Refer to KB Labs SOP No. 16, *Handling Complaints*.

### 11.2 DEPARTURES FROM PROCEDURES AND SPECIFICATIONS

It is the policy of KB Labs that documented procedures and standard specifications will be followed on a routine basis for all projects. However, the following exceptions may arise:

- If a client requests a departure from a documented procedure or standard specification, it will be noted in detail on the report to the client and in the project file.
- If KB Labs cannot, because of unexpected field conditions or operational circumstances, follow a documented procedure or a standard specification, the Field Chemist will immediate notify the Director of Operations and/or the Quality Assurance Officer, who

will then notify the client. The departure will be documented in the final report to the client and in the project file.

Refer to KB Labs' SOP No. 32, *Departures from Documented Policies and Procedures*.

**Table 11-1: Corrective Action**

<b><u>QC Activity</u></b>	<b><u>Acceptance Criteria</u></b>	<b><u>Recommended Corrective Action</u></b>
Initial Calibration	Follow protocol stated in reference method.	Rerun calibration. If necessary, prepare fresh calibration standards.
Method Blank	$\leq 1/10$ concentration in any sample associated with blank.	Re-prepare and reanalyze new method blank and samples associated with contaminated blank. If necessary, perform appropriate instrument maintenance.
GC/MS Tuning	BFB ion abundance criteria must be met as set forth in Method 8260b	Perform mass calibration. Retune hardware. If necessary, clean source.
Continuing Calibration	Follow protocol stated in reference method.	Rerun continuing calibration standard. If necessary, rerun initial standard calibration.
Surrogates	Within established control limits	Reanalyze samples that have one or more surrogates out of control.
MS/MSD Spikes	Within established control limits	Reanalyze samples that have one or more spikes out of control.
LCS	Within established control limits	

## 12.0 RECORD KEEPING AND DOCUMENT CONTROL

### 12.1 STORAGE OF PROJECT DATA

All **project data files** containing the items listed in Section 9.1 above are stored chronologically in a file cabinet in KB Labs' administrative office. No project data files are stored in the mobile laboratory units. In order to help maintain the integrity of data and to protect the confidentiality and proprietary rights of all clients, these files are archived as needed into banker's boxes that are kept in a secure storage area of the administrative office. All archived data are stored at least 5 years in accordance with NELAC standards. An access log will be maintained for retrieving the archived files. Access is limited to employees of KB Labs only. No data will be released to parties other than the client without written permission from the client.

Data from the analytical instrument computers are archived onto backup disks at least every six months. These disks are stored in the administrative office in fire proofs boxes in a designated area. They are labeled with the project number and project name and stored sequentially by project number.

Final reports to the client (see Section 9.3) are stored in electronic computer files by client and project name. These are regularly backed up on disk and stored in the administrative office fire proof boxes in a designated area. A photocopy of the final report to the client is also contained in the project management file, which is described below in Section 12.2

Refer to KB Labs SOP No. 008, *Filing and Archiving Project Records*.

### 12.2 STORAGE OF ADMINISTRATIVE RECORDS

**Project management files** are maintained by the Director of Operations in a separate file cabinet in the administrative office. These files are arranged alphabetically by client and within the client file, alphabetically by project name. A project management file generally contains the following documentation:

- Proposal or bid
- Contract
- Work Order
- Final Report
- Correspondence
- Notes

**Personnel files** are maintained alphabetically by the QA Officer in a separate file cabinet in the administrative office and contain the following documentation:

- Resume
- Training Record
- Demonstrations of Capability

**Mobile unit files** are maintained by the QA Officer in the administrative office. These files contain the following documentation:

- SOPs for analytical methods performed in the mobile unit
- MDL study data
- Copies of all demonstration of capability certifications
- PE sample data

### 12.3 MOBILE UNIT RECORD KEEPING

Each of the mobile units will maintain limited documentation for operations conducted in the unit. This documentation will include the following, each of which will be maintained in separate, labeled folders stored in a file cabinet:

- SOPs for analytical methods performed in the mobile unit
- Other company SOPs
- Most recent MDL study data for each method performed in the unit
- Copies of all demonstration of capability certifications for unit personnel
- PE sample data for at least the last three rounds for each analytical method performed
- Personnel Resume(s)
- Personnel Training Record (s)

In addition, each mobile unit will maintain a copy of the most recent version of the Laboratory Quality Manual and Health & Safety Manual and will maintain logbooks for sample receipt, instrument run, instrument maintenance and repair, standards, and refrigerator temperature. Completed logbooks will be stored in the administrative office in a designated area.

### 12.4 DOCUMENT CONTROL

Official KB Labs documentation such as the Laboratory Quality Manual, Laboratory Health & Safety Manual, SOPs, standard forms, etc. are updated annually or whenever necessary. It is important that the most recent revision of each document is utilized by all personnel. In order to facilitate this, each document will have the document file name, date of production, and revision number clearly indicated in the header or footer.

It is the responsibility of the Quality Assurance Supervisor to assure that all official documents are updated as required and that the latest revision is in use by all personnel. Copies of old outdated documentation will be kept on file by the QA Officer. A master list of the latest revision of all documentation will be maintained and posted in the administrative office by the Quality Assurance Supervisor.

Refer to KB Labs SOP No. 015, *Document Control*.

## 13.0 PREVENTIVE MAINTENANCE

To minimize the occurrence and severity of instrument failure, a preventive maintenance program for laboratory instruments has been implemented. The preventive maintenance performed for major pieces of analytical equipment is listed below in Table 13-1.

### 13.1 DOCUMENTATION OF ROUTINE MAINTENANCE AND NON-ROUTINE REPAIRS

All repairs are documented in the instrument logbook, which includes the date of maintenance or repair and description of work. Further documentation is provided in the instrument file, which includes complete documentation of repair work completed.

In the event of any instrument failure KB Labs will proceed with the following actions:

1. Repair of instrument by staff
2. Repair of instrument by service representative
3. Return instrument to place of manufacture for repair
4. Acquire new instrumentation

In the event of excessive down time, KB labs will either acquire instrumentation to complete project work or subcontract project work to fulfill project requirement.

### 13.2 LABORATORY SET-UP ROUTINE

Each time a mobile lab unit is relocated, the following setup routine is following:

1. Verify connection to generator fuel supply and trailer ground strap.
2. After starting generator, check voltage output. Voltage should be  $120 \pm 10$  volts.
3. Turn on climate control.
4. Turn on analytical instruments and allow heated zones to come to temperature. (After turning on GC/MS, pump down MSD (approximately 2 to 4 hours) to operating vacuum. Verified by the ion gauge controller.)
5. Check GC gas flow leaks settings. Check MS system for leaks if system will not pump down or if there is excessive noise in baseline.
6. Prepare reagent water blanks and analyze system blank. If blank passes, initial calibration can begin. (GC/MS must also pass tune check before sample analysis can begin.)

**Table 13-1: Preventive Maintenance Procedures**

<u>Procedure</u>	<u>Frequency</u>
<b>Gas Chromatograph</b>	
Change septa	When system develops leaks or as needed
Check carrier gas	Daily
Change carrier gas	When pressure falls below 100 psi
Cut edge of capillary column	When system performance declines
Change columns	When column performance declines or as needed
Change injector port liner	When dirty or as needed
<b>Mass Spectrometer</b>	
Backup system software	Monthly
Replace traps	Annually
Manufacturer's preventive maintenance	Annually
Clean source	When calibration compound criteria cannot be achieved or as needed
Keep instrument clean and dust-free	After each use
<b>Purge and Trap</b>	
Clean, bake, and purge spargers	Daily prior to use
Bake out trap	Daily prior to use
Replace trap	Quarterly or as needed
Replace fittings	Annually or as needed

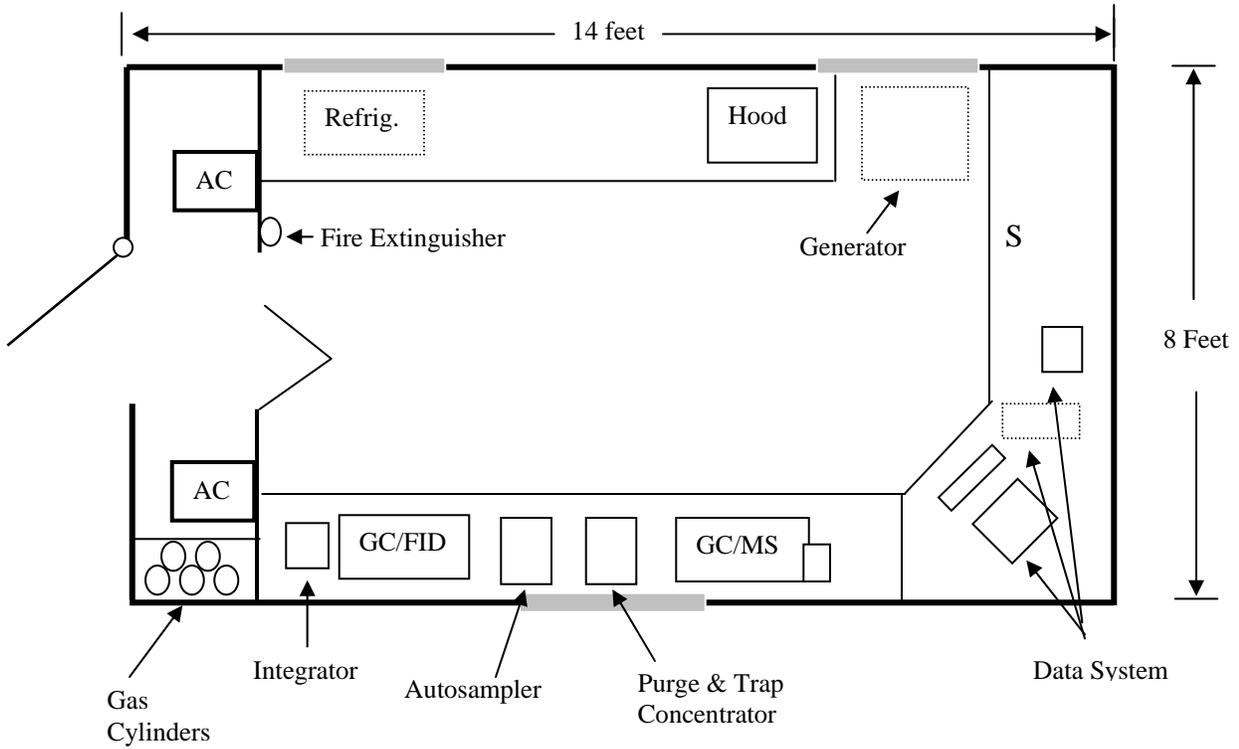
**Table 13-1: Preventive Maintenance Procedures (Cont'd)**

	<u>Procedure</u>	<u>Frequency</u>
<b>Support Equipment</b>		
Ovens, Refrigerators	Monitor temperature, keep units clean	Daily
Hot Plates	Keep units clean	After each use
Generator	Check oil level	Before starting
	Check battery fluid	Monthly
	Change oil and filter	Every 1000 hours
	Change air filter	Monthly, more often if conditions are dusty
	Check voltage	Upon startup

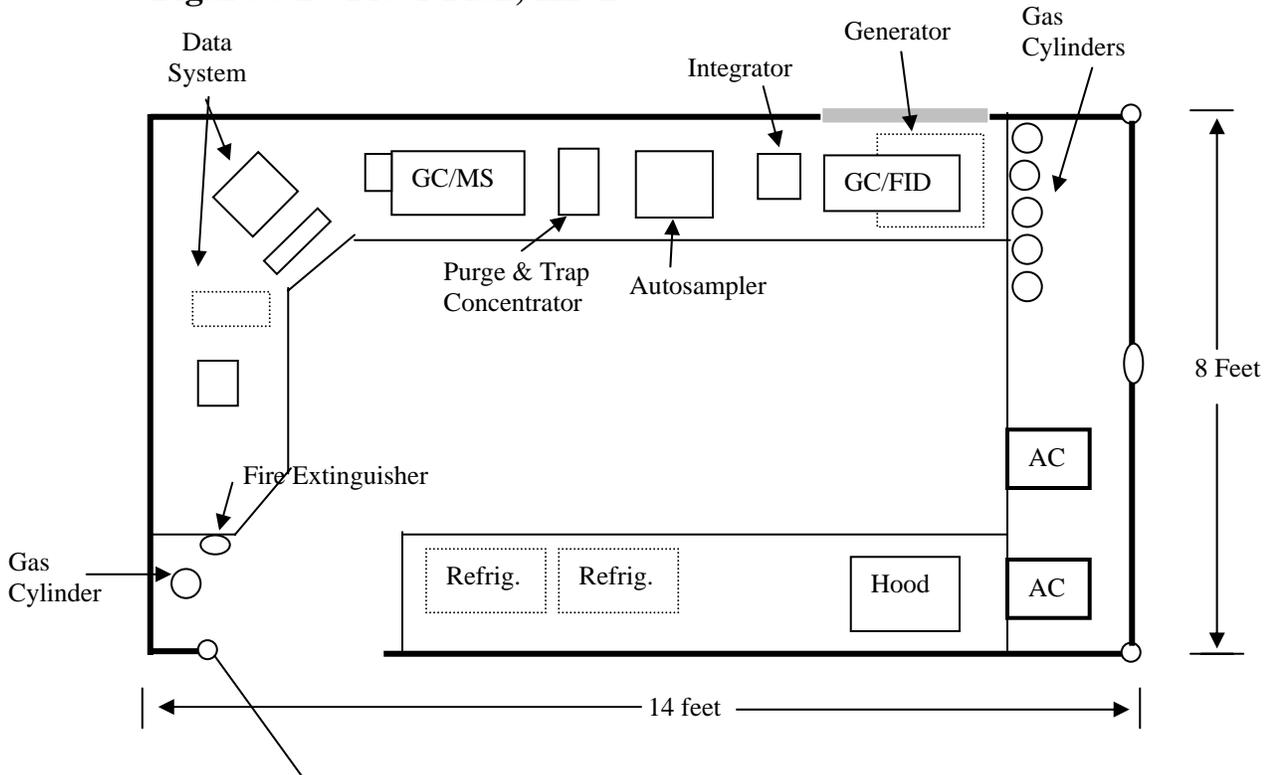
# **APPENDIX A**

## **Floor Plans**

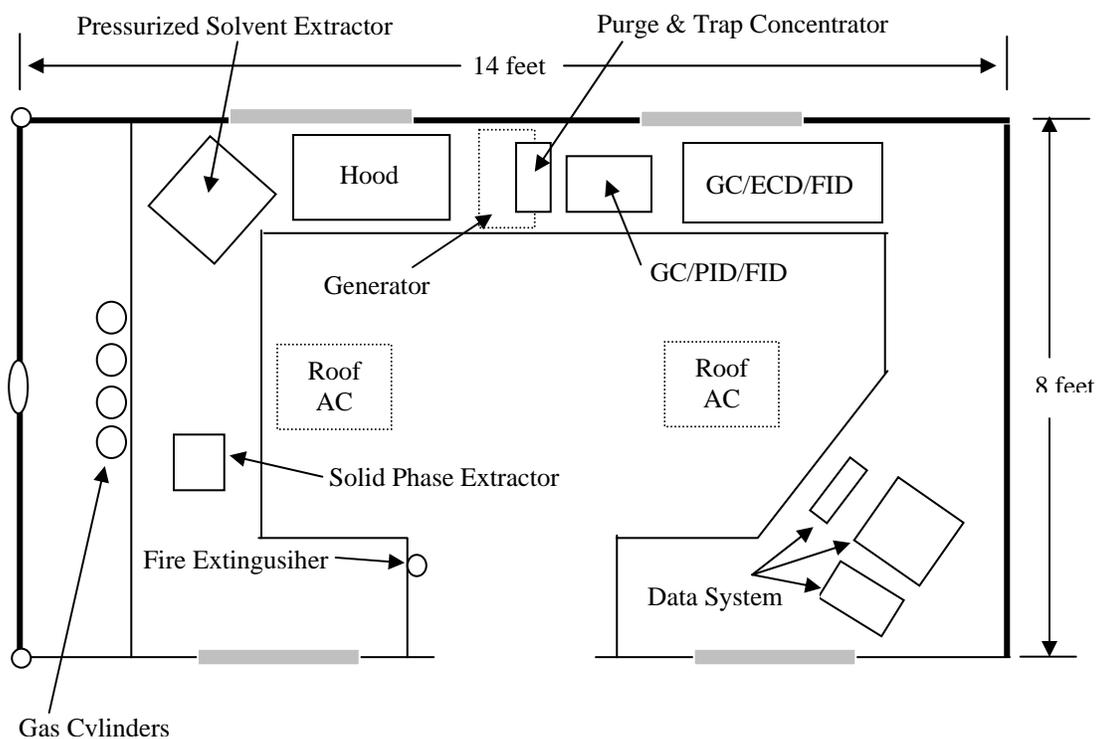
**Figure 3-1: Floor Plan, KB-1**



**Figure 3-2: Floor Plan, KB-2**



**Figure 3-3: Floor Plan, KB-3**



# **APPENDIX B**

## **Standard Forms**





## Demonstration of Capability Certification Statement

<b>Date:</b>	<b>Analyst Name:</b>	<b>Mobile Lab No:</b>
<b>Matrix:</b>	<b>Method Number:</b>	<b>SOP Number:</b>
<b>Parameters:</b>		

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

\_\_\_\_\_  
Bradley A. Weichert  
Technical Director

\_\_\_\_\_  
Date

\_\_\_\_\_  
Michael G. Winslow  
Quality Assurance Officer

\_\_\_\_\_  
Date



## Data Review Checklist

- \_\_\_\_\_ Check Data Folder Table of Contents Checklist
- \_\_\_\_\_ Check COCs vs. field data report
  - \_\_\_\_\_ Make sure all sample IDs match and are accounted for.
- \_\_\_\_\_ Data numbers vs field report
  - \_\_\_\_\_ Go through raw data page by page and compare numbers
  - \_\_\_\_\_ Highlight any numbers that need to be changed on the field report
- \_\_\_\_\_ Cover letter
- \_\_\_\_\_ Project narrative
  - \_\_\_\_\_ Change header box and review for accuracy (i.e. water description if waters are run)
  - \_\_\_\_\_ Change date at bottom
- \_\_\_\_\_ Run sequence/surrogate table
  - \_\_\_\_\_ Change header box
  - \_\_\_\_\_ Use daily sequence summaries to get sequence of samples
  - \_\_\_\_\_ Go through raw data to get surrogate recoveries
  - \_\_\_\_\_ Change date at bottom
- \_\_\_\_\_ Matrix spike table
  - \_\_\_\_\_ Change header box
  - \_\_\_\_\_ Comment section
  - \_\_\_\_\_ Change date at bottom
  - \_\_\_\_\_ Review recovery values
- \_\_\_\_\_ Final data report
  - \_\_\_\_\_ Significant figures (only two SF for dilutions)
  - \_\_\_\_\_ Put units on the table
- \_\_\_\_\_ Data report narrative
  - \_\_\_\_\_ Use highlighted preliminary field report
  - \_\_\_\_\_ If significant changes are necessary, inform Director of Operations
- \_\_\_\_\_ COCs
  - \_\_\_\_\_ If yellow pages are gone, client has them from the field; send photocopies of white pages.
  - \_\_\_\_\_ If yellows are still attached, send white pages and keep yellows as originals.

Signature: \_\_\_\_\_ Date: \_\_\_\_\_  
Title: Data Specialist

## Data Folder Table of Contents Checklist

- \_\_\_\_\_ 1. Disk with preliminary field report, FDEP report (if required)
- \_\_\_\_\_ 2. Work Order Form
- \_\_\_\_\_ 3. Field Logs
- \_\_\_\_\_ 4. COCs
- \_\_\_\_\_ 5. Preliminary field data report hardcopy
- \_\_\_\_\_ 6. Comment page (if required) detailing departures from method, problems, etc.
- \_\_\_\_\_ 7. Spike recovery summaries
- \_\_\_\_\_ 8. Initial calibration summaries (if performed)
- \_\_\_\_\_ 9. Tune record (MS only)
- \_\_\_\_\_ 10. Daily sequence summaries (any pertinent comments should be recorded here including reruns, dilutions, poor recoveries, data not used (incl. 'why'), etc.
- \_\_\_\_\_ 11. Analysis chromatograms and quantitation reports, in chronological order
- \_\_\_\_\_ 12. Screening chromatograms

Comments:

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Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Title: Data Review Specialist

## Annual System Audit Form

Date: \_\_\_\_\_ Auditor: \_\_\_\_\_

Mobile Lab No: \_\_\_\_\_ Analysis Requested: \_\_\_\_\_

Sample No.
Project Name:
Client Name:
Date Received:

	Yes	No
Was Chain of Custody properly filled in and signed?		
Does sample receipt logbook match the Chain of Custody?		
Were correct sample fractions received?		
Was the proper method chosen?		
Are bench sheets available for the analysis?		
Was analysis performed within holding times?		
Are all data calculations correct?		
Is there QC with the sample analysis batch?		
Was the project file reviewed?		
Does the instrument log date match the bench sheet date?		
Does the bench data match the data on the report?		
Were there any deficiencies noted?		
Are dilution factors documented?		
Is a run log included and complete?		
Is standard prep log complete?		

Comments	
Corrective Action	

# **APPENDIX C**

## **Final Report**

**KB LABS, INC.**  
6821 Southwest Archer Road  
Gainesville, Florida 32608

*telephone (352) 367-0073*  
*fax (352) 367-0074*

June 1, 2001

Paul Bunyan  
Big Boy Environmental, Inc.  
12345 Tall Guy Blvd  
Oxtown, FL 33333

**Re: Final Analytical Report, Big Ugly, Oxtown, FL**

Dear Mr. Bunyan:

Enclosed is the final report of the on-site analysis performed by KB Labs, Inc. at the Big Ugly site in Oxtown, FL. On-site analyses were performed May 23 – May 24, 2001. Included are a brief project narrative, tables listing quality control results, final analytical results, and sample chain-of-custody form. This information will also be sent electronically. Including this cover page, the Final Report includes eight pages.

KB Labs' mobile laboratories have been inspected by the FDOH Bureau of Laboratories and have been recommended for NELAP Certification as of April 1, 2003. Our personnel, methodology, proficiency testing, and quality assurance requirements complied with the guidelines of Chapter 64E-1 of the Florida Administrative Code and with the consensus standards adopted at the National Environmental Laboratory Accreditation Conference (NELAC). Data for the site referenced above were determined in accordance with published procedures under Test Methods for Evaluating Solid Waste (EPA SW-846, Update III Revised May 1997). Unless otherwise indicated on the quality control narrative accompanying the data report, the quality assurance and quality control procedures performed in conjunction with analysis of groundwater samples demonstrated that the reported data met our standards for accuracy and precision under NELAC Standards.

If you have any questions, please do not hesitate to call me or Kelly Bergdoll, President of KB Labs, at (352) 367-0073.

Sincerely,

KB Labs, Inc.

Todd Romero  
Director of Operations

## KB LABS, INC.

## PROJECT NARRATIVE

<b>Client:</b> Big Boy Environmental, Inc.	<b>Driller/Sampler:</b> Big Rig, Inc.	<b>Analyst:</b> M. Mathews
<b>Site:</b> Big Ugly, Oxtown, FL	<b>KB Project Manager:</b> Kelly Bergdoll	<b>KB Project No.</b> 0333
<b>Onsite Dates:</b> 5/23/01 – 5/24/01	<b>Client Project Manager:</b> Paul Bunyan	<b>Matrix:</b> Water

**Project Scope**

On May 23 – May 24, 2001, a total of eight (8) water samples were collected at the Big Ugly site in Oxtown, FL. Samples were analyzed onsite in the KB Labs mobile facility. The samples were analyzed for benzene, toluene, ethylbenzene, m&p-xylene, o-xylene, MTBE, naphthalene, and Diesel Range Organics (DRO).

**Analytical Procedure**

**VOCs** – All water samples were analyzed using SW846 Method 5030/8260 for waters. Ten (10) milliliters (mL) of water were purged with helium and the volatile organic compounds (VOCs) were collected on a solid-phase adsorption trap. The adsorption trap was heated and back-purged with helium and the components were separated by capillary column gas chromatography and measured with a mass spectrometer (GC/MS) operated in the electron impact full-scan mode. The individual VOCs in the samples were measured against corresponding VOC standards.

**DRO** – All water samples were first extracted in a vacuum extraction manifold using Sep-Pak C18 cartridges (2-gram). Sample volumes varied (20 – 200 mL) depending upon particulate content. The C18 cartridges were then solvent extracted with 5 mL of hexane. Samples extracts were then analyzed by gas chromatography/flame ionization detector (GC/FID). DRO in the samples was then measured against a corresponding diesel standard.

**Analytical Results**

Laboratory results were provided to the client on an as-completed or next-day basis. Final results of the on-site analyses are provided in a standard Excel spreadsheet format. The data produced and reported in the field has been reviewed and approved for this final report by the KB Labs Quality Assurance (QA) Officer.

**Quality Control (QC) Data**

**Surrogate Recoveries (VOCs only)** – Tables 1.1 – 1.2 list the daily analytical sequence and percent recovery results for surrogate compounds which were added to all analyses. Four (4) surrogate compounds were added to each analysis in order to continually monitor general method performance.

**Matrix Spike and Laboratory Control Spike Recoveries** – Table 2 lists the percent recovery results for matrix spike samples and /or laboratory control samples. A known amount of selected target compounds was added to selected field samples and/or to a laboratory blank sample in order to monitor the performance of the compounds in the actual matrix and in the laboratory blank sample.

**Method Blanks** – Daily analysis of laboratory reagent water samples was performed in order to monitor the cleanliness of the analytical system. No target compounds were detected on or above the reporting limits.

Signature: \_\_\_\_\_  
Title: Director of Operations

Date: June 1, 2001

**KB LABS, INC.**  
**6821 Southwest Archer Road**  
**Gainesville, Florida 32608**

*telephone (352) 367-0073*  
*fax (352) 367-0074*

**Table 1-1: VOC Analysis Sequence/Surrogate Percent Recoveries (5/23/01)**

<b>Client:</b> Big Boy Environmental, Inc.	<b>Driller/Sampler:</b> Big Rig, Inc.	<b>Analyst:</b> M. Mathews
<b>Site:</b> Big Ugly, Oxtown, FL	<b>KB Labs Project Manager:</b> Kelly Bergdoll	<b>KB Labs Project No:</b> 0333
<b>On-site Dates:</b> 5/23/01 - 5/24/01	<b>Client Project Manager:</b> Paul Bunyan	<b>Matrix:</b> Water

Station/Sample ID	Control Limits>>	S1* (80-120)	S2* (80 - 120)	S3* (80 - 120)	S4* (80 - 120)	
CCS 20 ug/L		81	98	97	89	
Method Blank		82	97	95	89	
SMP1		97	96	93	83	
SMP2		85	106	93	82	
SMP3		88	79	95	84	S2 low
SMP4		91	100	96	86	
SMP3 MS		90	94	93	84	
SMP3 MSD		86	95	93	85	
CCS 20 ug/L		85	100	94	89	

\* Surrogate Compounds

S1 = 1,2-Dichloroethane-D4

S2 = 1,4-Difluorobenzene

S3 = Toluene - D8

S4 = 4 - Bromofluorobenzene

Signature: \_\_\_\_\_

Title: Data Specialist

Date: June 1, 2001

**Table 1-2: VOC Analysis Sequence/Surrogate Percent Recoveries (5/24/01)**

<b>Client:</b> Big Boy Environmental, Inc.	<b>Driller/Sampler:</b> Big Rig, Inc.	<b>Analyst:</b> M. Mathews
<b>Site:</b> Big Ugly, Oxtown, FL	<b>KB Labs Project Manager:</b> Kelly Bergdoll	<b>KB Labs Project No:</b> 0333
<b>On-site Dates:</b> 5/23/01 - 5/24/01	<b>Client Project Manager:</b> Paul Bunyan	<b>Matrix:</b> Water

	<i>Control</i>	<b>S1*</b>	<b>S2*</b>	<b>S3*</b>	<b>S4*</b>	
<b>Station/Sample ID</b>	<i>Limits&gt;&gt;</i>	<i>(80-120)</i>	<i>(80 - 120)</i>	<i>(80 - 120)</i>	<i>(80 - 120)</i>	
CCS 20 ug/L		91	101	92	87	
Method Blank		79	95	96	88	S1 low
REF/LCS		121	103	94	84	S1 high
SMP5		80	96	91	83	
SMP6		120	115	95	93	
SMP7		83	93	97	89	
SMP8		80	95	98	88	
CCS 20 ug/L		119	100	90	82	

**\* Surrogate Compounds**

S1 = 1,2-Dichloroethane-D4

S2 = 1,4-Difluorobenzene

S3 = Toluene - D8

S4 = 4 - Bromofluorobenzene

Signature: \_\_\_\_\_

Title: Data Specialist

Date: June 1, 2001

**Table 2: VOC Spike Compound Percent Recoveries**

<b>Client:</b> Big Boy Environmental	<b>Driller/Sampler:</b> Big Rig, Inc.	<b>Analyst:</b> M. Mathews
<b>Site:</b> Big Ugly, Oxtown, FL	<b>KB Labs Project Manager:</b> Kelly Bergdoll	<b>KB Labs Project No:</b> 0333
<b>On-site Dates:</b> 5/23/01 - 5/24/01	<b>Client Project Manager:</b> Paul Bunyan	<b>Matrix:</b> Water

<b>Spike Compounds * &gt;&gt;</b>	<b>VOC1</b>	<b>VOC2</b>	<b>VOC3</b>	<b>VOC4</b>	<b>VOC5</b>	<b>VOC6</b>	<b>VOC7</b>	<b>VOC8</b>				<b>Comment</b>
<i>Control Limits ** &gt;&gt;</i>	70-130	75-119	79-114	81-114	74-120	78-116	70-130	70-130				
<i>Warning Limits** &gt;&gt;</i>	80-120	82-111	85-108	86-108	82-113	85-110	80-120	80-120				
<i>RPD Limit &gt;&gt;</i>	20	20	20	20	20	20	20	20				
<b>Station/Sample ID:</b>												
SMP3 MS	77	91	98	94	92	93	100	97				
SMP3 MSD	85	92	99	94	91	93	116	91				
RPD	10	1	1	0	1	0	15	6				
REF/LCS	NA	95	97	92	96	99	120	111				

\*\* Control limits based historical matrix spike recoveries.

\* Spike Compounds

- VOC1 = MTBE
- VOC2 = Benzene
- VOC3 = Toluene
- VOC4 = Ethylbenzene
- VOC5 = m&p-Xylene
- VOC6 = o-Xylene

- VOC7 = Naphthalene
- VOC8 = Diesel

Signature: \_\_\_\_\_

Title: Data Specialist

Date: June 1, 2001

**Analytical Data**  
**Big Ugly, Oxtown, FL**  
**5/23/01 – 5/24/01**

	Reporting Limits (ug/L)	SMP1	SMP2	SMP3	SMP4	SMP5	SMP6	SMP7	SMP8				
MTBE	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Benzene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Toluene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Ethylbenzene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
m&p-Xylene	<1	<1	1.5	<1	<1	<1	<1	<1	<1				
o-Xylene	<1	<1	<1	<1	<1	<1	<1	<1	<1				
Naphthalene	<1	<1	<1	<1	1.7	<1	<1	2.8	<1				
DRO (mg/L)	See Note	<2.0	<2.9	<2.0	<4.3	<2.0	<4.0	<4.3	<5.0				

Note: DRO reporting limits vary with the total volume of sample processed.  
This volume will vary depending the particulate content of the sample.

Signature: \_\_\_\_\_

Title: Data Specialist

Date: 6/1/01

**Sample Receipt and Acceptance**

- 1.0 KB Labs will supply pre-cleaned and certified sample containers to the client as required.
- 2.0 The Field Chemist will initiate and release to the client a Chain-of-Custody (COC) Record at the time the sample containers are released.
- 3.0 The Field Chemist will receive samples from a member of the client field sampling team and is the designated sample custodian.
- 4.0 When samples are received, the Field Chemist will determine the following:
  - 4.1 Whether samples have been received on ice.
  - 4.2 Whether the samples are in the appropriate sample containers. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.3 Whether the samples are of adequate volume or mass.
  - 4.4 Whether there are signs of leakage, breakage, or contamination.
  - 4.5 Whether headspace is present in the sample container (VOCs only).
  - 4.6 Whether proper preservation (if any) has been added. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.7 Whether holding times have been met. Refer to Table 6-1 of the Laboratory Quality Manual.
  - 4.8 Whether samples are properly labeled. Proper labeling will include the following:
    - The use of indelible ink.
    - The use of durable labels that are not easily removed.
    - Writing that can be clearly read and understood.
  - 4.9 Whether the COC Record has all the necessary and proper documentation. This will include the following:
    - Identification of samples
    - Location of sample collection
    - Date and time of sample collection
    - Sample type
    - Any special comments concerning the sample
    - Signatures of field sampler and sample custodian/field chemist

- 4.10 All samples received from the client must be recorded on the COC regardless of what the client says.
- 5.0 If any of the above conditions are not met, the Field Chemist will note the improper conditions for each sample on the COC Record, and he will immediately notify the field team leader of these improper conditions.
- 6.0 The field team leader must then make the decision whether to reject the samples in question.
- 7.0 Only after verifying the status of the samples, the Field Chemist will sign the Chain-of-Custody (COC) Record.
- 8.0 The Field Chemist will place the third copy (pink) of the COC Record into the sample receipt logbook.
- 9.0 The Field Chemist will return the second (yellow) copy to the client field team leader.
- 10.0 The Field Chemist will retain the original (white) copy of the COC Record with the project data file.



**KB LABS, INC.**

**Table 1: Analytical Run Sequence/Surrogate Percent Recoveries**

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

Sample ID	Date of Analysis	Surrogate % Recovery				Surrogate Control Limits			
		S1*	S2*	S3*	S4*	S1*	S2*	S3*	S4*
Example		99	102	105	98	Pass	Pass	Pass	Pass
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
						< LCL	< LCL	< LCL	< LCL
<b>Comments:</b>		Although some surrogates may be out of the control percent recovery range, other supporting QC, such as matrix spikes, matrix spike duplicates, method blanks, and laboratory control samples, are performed by KB Labs to further validate reported data.							

**\*Surrogate Compounds:**

S1 = Dibromofluoromethane (54% - 149%)

S2 = 1,2- Dichloroethane-D4 (61% - 156%)

S3 = Toluene-D8 (72% - 127%)

S4 = 4-Bromofluorobenzene (72% - 125%)

KB LABS, INC.

Table 2: VOC Spike Compound Percent Recoveries

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No.:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

**Matrix Spike/Matrix Spike Duplicate (MS/MSD):**

Matrix Spike Compounds	Date of Analysis:								
	Control Limits			Percent Recoveries			Control Limit Checks		
	Lower	Upper	RPD	MS	MSD	RPD	MS	MSD	RPD
Dichlorodifluoromethane	30	163	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dichloromonofluoromethane	53	156	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloromethane	34	142	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Vinyl Chloride	40	143	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromomethane	30	147	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloroethane	30	160	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Trichlorofluoromethane	45	154	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Freon 113	50	143	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1-Dichloroethene	45	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Methylene Chloride	49	148	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
trans-1,2-Dichloroethene	47	151	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
MtBE	41	153	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1-Dichloroethane	51	142	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
2,2-Dichloropropane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
cis-1,2-Dichloroethene	59	156	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chloroform	65	139	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Carbon Tetrachloride	47	150	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Benzene	65	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Trichloroethene	70	136	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Toluene	79	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,1-Trichloroethane	54	146	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichloroethane	71	147	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichloropropane	64	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dibromomethane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromodichloromethane	50	166	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
c-1,3-Dichloropropene	52	168	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
t-1,3-Dichloropropene	71	152	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,2-Trichloroethane	61	150	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dibromoethane	59	157	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Dibromochloromethane	58	157	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Chlorobenzene	79	121	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,1,2-Tetrachloroethane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Tetrachloroethene	57	152	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3-Dichloropropane	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Ethylbenzene	74	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
m,p-Xylene	73	132	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
o-Xylene	74	129	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Styrene	74	123	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromoform	42	149	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Isopropylbenzene	76	131	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,1,2,2-Tetrachloroethane	53	144	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Bromobenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
n-Propylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
2-Chlorotoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
4-Chlorotoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3,5-Trimethylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
tert-Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,4 Trimethylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
sec Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
p-Isopropyltoluene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,3-Dichlorobenzene	77	122	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,4-Dichlorobenzene	79	120	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dichlorobenzene	78	121	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
n-Butylbenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2-Dibromo-3-chloropropan	36	161	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,4-Trichlorobenzene	57	154	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Hexachlorobutadiene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
Naphthalene	47	158	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!
1,2,3-Trichlorobenzene	70	130	20	0	0	#DIV/0!	< LCL	< LCL	#DIV/0!

**Note:** Control Limits are based on a semi-annual historical evaluation of mobile unit.

**KB LABS, INC.**

**Table 2: VOC Spike Compound Percent Recoveries**

<b>Client:</b> Tetra Tech NUS	<b>Driller/Sampler:</b>	<b>Analyst:</b>
<b>Site:</b> NAS Jacksonville	<b>KB Labs Project Manager:</b> Todd Romero	<b>KB Labs Project No.:</b>
<b>On-site Dates:</b>	<b>Client Project Manager:</b>	<b>Matrix:</b> Water

**Laboratory Control Spikes (LCS):**

Spike Compounds	Control Limits		Percent Recoveries			Control Limit Checks		
	Lower	Upper	LCS#1	LCS#2	LCS#3	LCS#1	LCS#2	LCS#3
	Dichlorodifluoromethane	43	to 178	0	0	0	< LCL	< LCL
Chloromethane	42	to 176	0	0	0	< LCL	< LCL	< LCL
Vinyl Chloride	28	to 161	0	0	0	< LCL	< LCL	< LCL
Bromomethane	31	to 157	0	0	0	< LCL	< LCL	< LCL
Chloroethane	44	to 162	0	0	0	< LCL	< LCL	< LCL
Trichlorofluoromethane	46	to 167	0	0	0	< LCL	< LCL	< LCL
Freon 113	59	to 168	0	0	0	< LCL	< LCL	< LCL
1,1-Dichloroethene	43	to 156	0	0	0	< LCL	< LCL	< LCL
Methylene Chloride	43	to 160	0	0	0	< LCL	< LCL	< LCL
trans-1,2-Dichloroethene	39	to 169	0	0	0	< LCL	< LCL	< LCL
MtBE	46	to 157	0	0	0	< LCL	< LCL	< LCL
1,1-Dichloroethane	43	to 156	0	0	0	< LCL	< LCL	< LCL
2,2-Dichloropropane	70	to 130	0	0	0	< LCL	< LCL	< LCL
cis-1,2-Dichloroethene	60	to 150	0	0	0	< LCL	< LCL	< LCL
Chloroform	60	to 145	0	0	0	< LCL	< LCL	< LCL
Carbon Tetrachloride	43	to 154	0	0	0	< LCL	< LCL	< LCL
Benzene	63	to 129	0	0	0	< LCL	< LCL	< LCL
Trichloroethene	47	to 164	0	0	0	< LCL	< LCL	< LCL
Toluene	78	to 128	0	0	0	< LCL	< LCL	< LCL
1,1,1-Trichloroethane	52	to 152	0	0	0	< LCL	< LCL	< LCL
1,2-Dichloroethane	68	to 147	0	0	0	< LCL	< LCL	< LCL
1,2-Dichloropropane	53	to 159	0	0	0	< LCL	< LCL	< LCL
Dibromomethane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Bromodichloromethane	45	to 170	0	0	0	< LCL	< LCL	< LCL
c-1,3-Dichloropropene	50	to 174	0	0	0	< LCL	< LCL	< LCL
t-1,3-Dichloropropene	78	to 167	0	0	0	< LCL	< LCL	< LCL
1,1,2-Trichloroethane	67	to 149	0	0	0	< LCL	< LCL	< LCL
1,2-Dibromoethane	65	to 153	0	0	0	< LCL	< LCL	< LCL
Dibromochloromethane	68	to 151	0	0	0	< LCL	< LCL	< LCL
Chlorobenzene	79	to 128	0	0	0	< LCL	< LCL	< LCL
1,1,1,2-Tetrachloroethane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Tetrachloroethene	60	to 151	0	0	0	< LCL	< LCL	< LCL
1,3-Dichloropropane	70	to 130	0	0	0	< LCL	< LCL	< LCL
Ethylbenzene	77	to 125	0	0	0	< LCL	< LCL	< LCL
m,p-Xylene	77	to 134	0	0	0	< LCL	< LCL	< LCL
o-Xylene	79	to 125	0	0	0	< LCL	< LCL	< LCL
Styrene	76	to 124	0	0	0	< LCL	< LCL	< LCL
Bromoform	52	to 148	0	0	0	< LCL	< LCL	< LCL
Isopropylbenzene	87	to 138	0	0	0	< LCL	< LCL	< LCL
1,1,2,2-Tetrachloroethane	59	to 143	0	0	0	< LCL	< LCL	< LCL
Bromobenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
n-Propylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
2-Chlorotoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
4-Chlorotoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,3,5-Trimethylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
tert-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2,4-Trimethylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
sec-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
p-Isopropyltoluene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,3-Dichlorobenzene	84	to 117	0	0	0	< LCL	< LCL	< LCL
1,4-Dichlorobenzene	84	to 122	0	0	0	< LCL	< LCL	< LCL
1,2-Dichlorobenzene	85	to 116	0	0	0	< LCL	< LCL	< LCL
n-Butylbenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2-Dibromo-3-chloropropane	43	to 169	0	0	0	< LCL	< LCL	< LCL
1,2,4-Trichlorobenzene	63	to 149	0	0	0	< LCL	< LCL	< LCL
Naphthalene	52	to 150	0	0	0	< LCL	< LCL	< LCL
Hexachlorobutadiene	70	to 130	0	0	0	< LCL	< LCL	< LCL
1,2,3-Trichlorobenzene	70	to 130	0	0	0	< LCL	< LCL	< LCL

**Note:** Control limits are based on method guidance.