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NAS WHITING FIELD  
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SAMPLING AND ANALYSIS PLAN FOR REMEDIAL INVESTIGATION ADDENDUM FOR  
OPERABLE UNIT 25 (OU 25) SITE 40 BASEWIDE GROUNDWATER NAS WHITING FIELD FL  
6/1/2010  
TETRA TECH

# Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



Rev. 1  
June 2010

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

### Remedial Investigation Addendum for Site 40, Basewide Groundwater Operable Unit 25

Naval Air Station Whiting Field  
Milton, Florida

Contract Task Order 0064

June 2010



NAS Jacksonville  
Jacksonville, Florida 32212-0030

**SAP WORKSHEET #1--TITLE PAGE**  
(UFP-QAPP Manual Section 2.1)

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

**JUNE 2010**

**Remedial Investigation Addendum**  
**Site 40, Basewide Groundwater**  
**Operable Unit (OU) 25**  
**Naval Air Station (NAS) Whiting Field**  
**Milton, Florida**

**Prepared for:**  
**Naval Facilities Engineering Command**  
**Southeast**  
**Building 903**  
**NAS Jacksonville**  
**Jacksonville, Florida 32212-0030**

**Prepared by:**  
**Tetra Tech NUS, Inc.**  
**661 Andersen Drive, Foster Plaza 7**  
**Pittsburgh, Pennsylvania 15220-2745**

**Prepared under:**  
**Comprehensive Long-Term Environmental Action Navy**  
**Contract Number N62467-04-D-0055**  
**Contract Task Order 0064**

**Project-Specific Sampling and Analysis Plan**  
**Site Name/Project Name:** Site 40, Basewide Groundwater  
**Site Location:** NAS Whiting Field, Milton, Florida

**Title:** Data Gaps Remedial Investigation Addendum Work Plan  
**Revision Number:** 1  
**Revision Date:** June 2010

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

**Document Title:** Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan),  
June 2010, Site 40, Basewide Groundwater Remedial Investigation Addendum, Naval Air  
Station (NAS) Whiting Field, Milton, Florida

**Lead Organization:** Naval Facilities Engineering Command Southeast (NAVFAC SE)  
**Preparer's Name and Organizational Affiliation:** Katie Newman, Tetra Tech NUS, Inc.  
**Preparer's Address and Telephone Number:** 1558 Village Square Blvd., Suite 2  
Tallahassee, Florida 32309  
(850) 385-9899

**Preparation Date (Day/Month/Year):** June, 16 2010



Investigative Organization's Project Manager:

Signature/Date – June 16, 2010  
Rich May, Tetra Tech NUS, Inc.

Investigative Organization's Project Quality  
Assurance Manager:



Signature/Date – June 16, 2010  
Kelly Carper  
Tetra Tech NUS, Inc.

Lead Organization's Project Manager:

Signature/Date  
Benjamin "Tread" Kissam  
Naval Facilities Engineering Command Southeast  
TUCKER.JONATHAN.P. Digitally signed by TUCKER.JONATHAN.P.1239524180  
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI,  
ou=USN, cn=TUCKER.JONATHAN.P.1239524180  
Date: 2010.07.02 13:17:35 -04'00'  
1239524180

Navy Quality Assurance Officer:

Signature/Date  
Navy Chemist/Quality Assurance Officer  
Naval Facilities Engineering Command Atlantic

Approval Signatures:

Signature/Date  
Craig Benedikt  
U.S. Environmental Protection Agency Region 4

Signature/Date  
John Winters  
Florida Department of Environmental Protection

## EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) encompasses Field Sampling Plan and Quality Assurance Project Plan (QAPP) requirements for a Remedial Investigation (RI) Addendum Work Plan at Site 40 - Basewide Groundwater (Site 40) at Naval Air Station (NAS) Whiting Field, Milton, Florida. This document constitutes the planning document, addressing specific protocols for sample collection, sample handling and storage, chain-of-custody, laboratory and field analyses, data validation, and data reporting.

This SAP has been prepared by Tetra Tech NUS, Inc. (Tetra Tech) on behalf of Naval Facilities Engineering Command Southeast (NAVFAC SE) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62467-04-D-0055, Contract Task Order (CTO) 0064. This SAP was generated for and complies with applicable United States Department of the Navy, United States Environmental Protection Agency (USEPA) Region 4, and Florida Department of Environmental Protection (FDEP) requirements, regulations, guidance, and technical standards. This includes the Department of Defense (DoD), Department of Energy (DOE), and USEPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. To comply with IDQTF requirements, this 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).

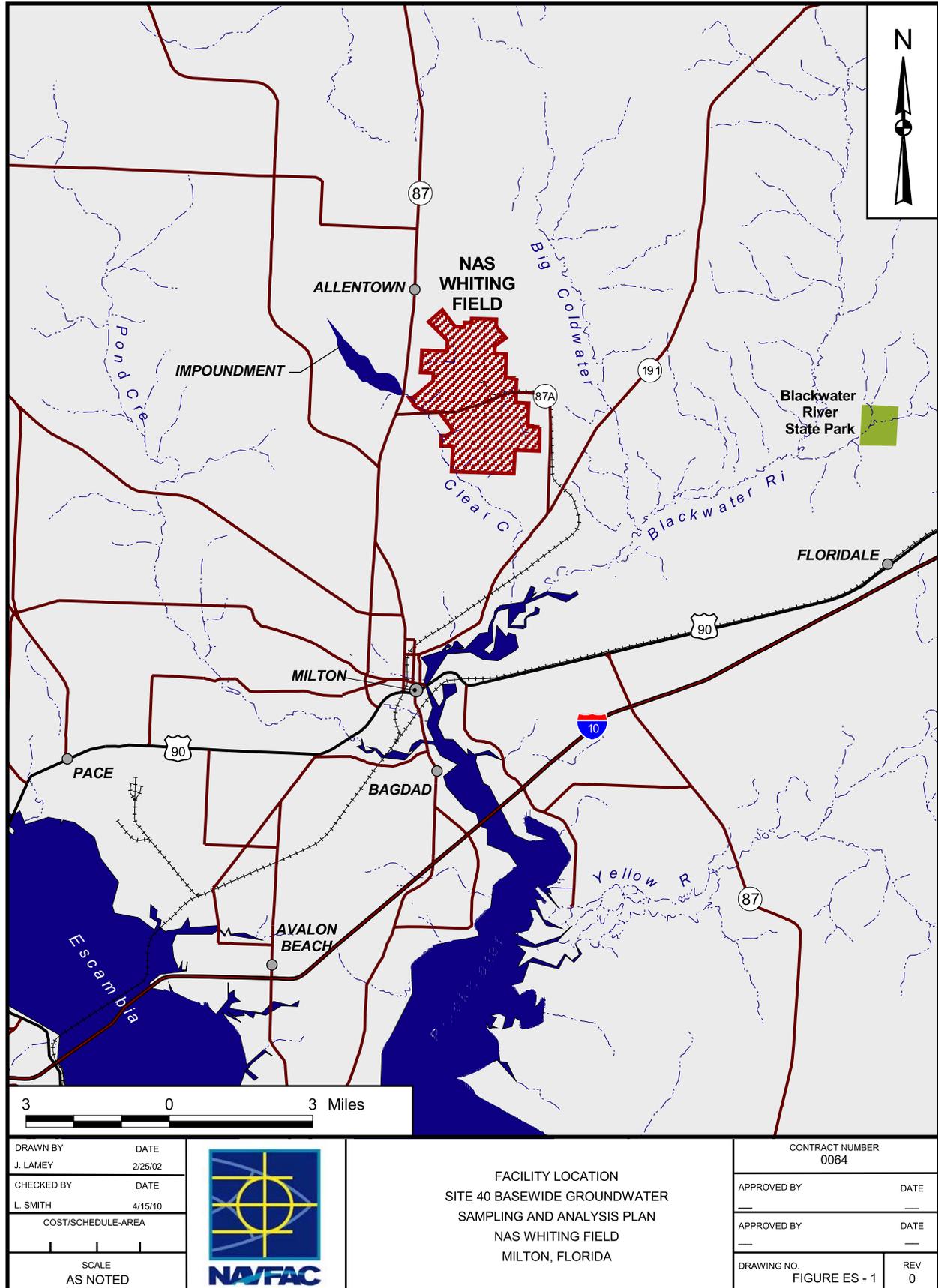
NAS Whiting Field is located in Santa Rosa County, in Florida's northwest coastal area, approximately 5.5 miles north of Milton and 25 miles northeast of Pensacola, as identified on Figure ES-1. NAS Whiting Field is 3,842 acres in size, and consists of two airfields (North Field and South Field) separated by a centralized industrial area. The centralized industrial area is separated, for the purposes of the report, into the North Field Industrial Area which supports North Field and fixed-wing aircraft and the South Field Industrial Area supports South Field and helicopter training. The locations of all Installation Restoration Program (IRP) sites and selected underground storage tank (UST) sites at NAS Whiting Field are identified on the Site Location Map on Figure ES-2.

In 1997, the Project Team designated Site 40, also known as Operable Unit (OU) 25, as a separate site to simplify addressing commingled groundwater plumes underlying multiple Installation Restoration (IR) sites. Site 40 was subsequently expanded to include all groundwater underlying the NAS Whiting Field. As a result, remedial activities for soil at all IR sites are addressed separately from groundwater.

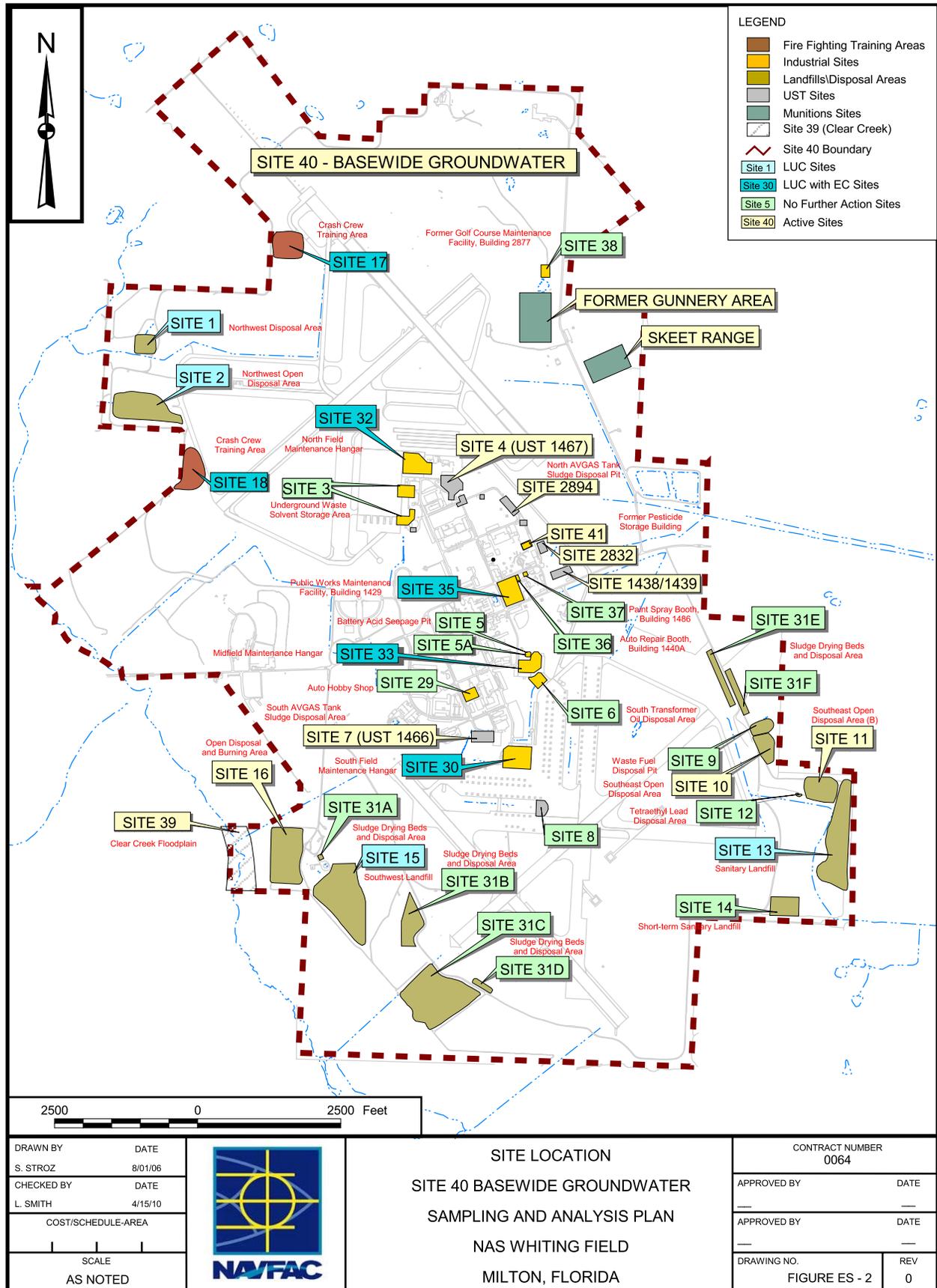
In 2007, the Project Team determined the groundwater analytical data set developed in 2000 required new data that will be used to supplement the Site 40 RI Report. Three basewide groundwater sampling events were planned to provide trending data. However, a data gap became apparent in 2008 when 20 shallow wells on the eastern side of the North Field plume were found to be perched above the water

table. A subsequent review of rainfall data revealed a regional drought began in approximately 2000 and continued until 2008. As a result of the drought, the water table in the North Field dropped an average of 8 feet and 20 shallow monitoring wells no longer extended to the water table, but were perched above the water table and were dry, or providing water that did not reflect conditions of the surficial aquifer, and therefore were not usable. The Project Team has determined that new wells are required to complete delineation of the eastern boundary of the North Field Plume.

The primary objective of this UFP-SAP is to gather additional data to address the delineation gap for the eastern area of the North Field plume and to add this data to the previously collected Site 40 RI data such that the Site 40 Basewide Groundwater RI Report and Feasibility Study can be prepared. The effort will include installing and sampling nine new shallow monitoring wells to redefine the eastern boundary of the North Field plume. In addition, 10 existing monitoring wells will be sampled at Sites 4, 2894, and 1438/1439 to address minor data gaps by acquiring supplemental data that is representative of current conditions.



P:\GIS\WHITINGFIELD\_NAS\APR\RI\_SITE\_MAPS.APR FACILITY LOCATION 4/15/10 SP



P:\GIS\WHITINGFIELD\_NAS\APR\SITE\_STATUS\_MAP.APR UPDATED SITE STATUS MAP 4/15/10 SP ARCHIVE DVD

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## ACRONYMS AND ABBREVIATIONS

°C	Degrees Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
ABB-ES	Asea Brown Boveri-Environmental Services
AES	Atomic Emission Spectroscopy
AVGAS	Aviation Gasoline
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BTEX	Benzene, Toluene, Ethylbenzene, and Total Xylenes
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
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
DO	Dissolved Oxygen
DoD	Department of Defense
DOE	Department of Energy
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
Empirical	Empirical Laboratories, LLC
Ext	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FOL	Field Operations Leader

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

FS	Feasibility Study
FTMR	Field Task Modification Request
GAC	Granular Activated Carbon
GC/MS	Gas Chromatograph/Mass Spectrometer
GCTL	Groundwater Clean-up Target Level
GIR	General Information Report
GIS	Geographic Information System
HCl	Hydrochloric Acid
HNO <sub>3</sub>	Nitric Acid
HASP	Health and Safety Plan
HSM	Health and Safety Manager
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
IR	Installation Restoration
IRP	Installation Restoration Program
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOQ	Limit of Quantitation
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
mL	Milliliter
MPC	Measurement Performance Criterion
MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	Mean Sea Level
NA	Not Applicable
NAS	Naval Air Station
NAVFAC SE	Naval Facilities Engineering Command Southeast

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

NPL	National Priorities List
NTU	Nephelometric Turbidity Unit
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PAL	Project Action Limit
PM	Project Manager
PoC	Point of Contact
PPE	Personal Protective Equipment
PQO	Project Quality Objective
PT	Proficiency Testing (previously known as performance evaluation [PE] sample)
PVC	Polyvinyl Chloride
PWC	Public Works Center
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
r	Linear Regression Correlation Coefficient
r <sup>2</sup>	Coefficient of Determination
RF	Response Factor
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SAR	Site Assessment Report
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language

## ACRONYMS AND ABBREVIATIONS (CONTINUED)

SRCR	Sample Receipt Condition Report
SSO	Site Safety Officer
SVE	Soil Vapor Extraction
TBD	To Be Determined
TCE	Trichloroethene
TCE+	Daughter products: cis- and trans-1,2-dichloroethylene and vinyl chloride
Tetra Tech	Tetra Tech NUS, Inc.
UFP	Uniform Federal Policy
µg/L	Micrograms per Liter
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VC	Vinyl Chloride
VOC	Volatile Organic Compound

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

**Site Name/Number:** (NAS Whiting Field, Milton, Florida)  
**Operable Unit (OU):** OU 25 - Site 40, Basewide Groundwater  
**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) 0064

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* [United States Environmental Protection Agency (USEPA), 2005] and the *USEPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS* (USEPA, 2002).
2. Identify regulatory program: Florida Department of Environmental Protection (FDEP) Petroleum Contamination Site Cleanup process [Chapter 62-770 of the Florida Administrative Code (F.A.C.)] Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).
3. This SAP is a project-specific SAP.
4. List dates of scoping sessions that were held:

<u>Scoping Session</u>	<u>Date</u>
Data Quality Objective (DQO) Scoping Meeting (Project Team)	August 26, 2009
5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

<u>Title</u>	<u>Date</u>
Draft Site 40 Remedial Investigation, Tetra Tech NUS, Inc.	April 2004
Remedial Investigation/Feasibility Study (RI/FS) General Information Report (GIR), NAS Whiting Field- Asea Brown Boveri-Environmental Services (ABB-ES)	1998
6. List organizational partners (stakeholders) and connection with lead the organization:  
USEPA Region IV – Regulatory Stakeholder  
FDEP – Regulatory Stakeholder
7. Lead organization:  
NAVFAC SE – Property Owner
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)

<b>Name of SAP Recipients</b>	<b>Title/Role</b>	<b>Organization</b>	<b>Telephone Number</b>	<b>E-mail Address or Mailing Address</b>	<b>Document Control Number</b>
Tread Kissam	Navy Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC SE Building 903 NAS Jacksonville Jacksonville, FL 32212	(904) 542-6826	benjamin.kissam@navy.mil	NA
Mike Pattison	Installation Restoration Program (IRP) Manager/ NAS Whiting Field Point of Contact (POC)	Public Works Department (PWD) NAS Whiting Field 7183 Langley St. Bldg. 1416 Milton, FL 32570-6159	(850) 623-7268	michael.pattison@navy.mil	NA
Bonnie Capito (copy of final cover letter only)	Administrative Record / Librarian and Records Manager	NAVFAC LANT HQ Code EV32 1510 Gilbert Street Norfolk, VA 23511-2699	(757) 322-4785	bonnie.capito@navy.mil	NA
Craig Benedikt	USEPA RPM/ Provides Regulator Input	U.S. Environmental Protection Agency Region IV Superfund Division FWD-Federal Facilities Branch 61 Forsyth Street SW Atlanta, GA 30303-8960	(404) 562-8555	benedikt.craig@epamail.epa.gov	NA
John Winters	FDEP RPM/ Provides Regulator Input	Remedial Project Manager Florida Department of Environmental Protection 2600 Blair Stone Road Mail Station 4535 Tallahassee, FL 32399-2400	(850) 245-8999	john.winters@dep.state.fl.us	NA

<b>Name of SAP Recipients</b>	<b>Title/Role</b>	<b>Organization</b>	<b>Telephone Number</b>	<b>E-mail Address or Mailing Address</b>	<b>Document Control Number</b>
Debra Humbert (distribution letter only)	Program Manager / Manages Navy Initiatives	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-1990	debra.humbert@tetrattech.com	NA
Chris Pike (distribution letter only)	Deputy Program Manager/ Manages Program Activities	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8861	chris.pike@tetrattech.com	NA
Richard May	Project Manager (PM)/ Manages Project Activities	Tetra Tech NUS, Inc. 1558 Village Square Blvd., Suite 2 Tallahassee, FL 32309	(850) 385-9899 Ext. 1356	richard.may@tetrattech.com	NA
Larry Smith	Field Operations Leader (FOL)/ Site Safety Officer (SSO)/ Manages Field Operation and Site Safety Issues	Tetra Tech NUS, Inc. 1558 Village Square Blvd., Suite 2 Tallahassee, FL 32309	(850) 385-9899 Ext. 1360	larry.smith@tetrattech.com	NA
Kelly Carper (electronic copy only)	Quality Assurance Manager (QAM)/ Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8615	kelly.carper@tetrattech.com	NA
Matt Soltis [Health and Safety Plan (HASP) only]	Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8912	matt.soltis@tetrattech.com	NA

<b>Name of SAP Recipients</b>	<b>Title/Role</b>	<b>Organization</b>	<b>Telephone Number</b>	<b>E-mail Address or Mailing Address</b>	<b>Document Control Number</b>
Matthew Kraus (electronic copy only)	Project Chemist/ Provides Coordination with Laboratory(s)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8729	matthew.kraus@tetrattech.com	NA
Joseph Samchuck (electronic copy only)	Data Validation Manager (DVM)/ Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8510	joseph.samchuck@tetrattech.com	NA
Lee Leck (electronic copy only)	Data Manager/ Manages Databases	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	(412) 921-8856	lee.leck@tetrattech.com	NA
Kim Kostzer (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical Laboratories, LLC (Empirical) 621 Mainstream Drive, Suite 270 Nashville, TN 37228	(615) 345-1115	kkostzer@empirilabs.com	NA
Driller – To Be Determined (TBD) (electronic copy only)	Well Installation Subcontractor PM/ Provides Drilling Services	TBD	TBD	TBD	NA
Surveyor (TBD) (electronic copy only)	Surveyor/Provides Surveying Services	TBD	TBD	TBD	NA
Investigation- Derived Waste (IDW) Subcontractor (electronic copy only)	IDW Subcontractor/ Provides IDW Services	TBD	TBD	TBD	NA

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

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

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

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

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
<b>Navy and Regulator Project Team Personnel</b>					
Tread Kissam	Navy/ RPM/ Manages Project Activities for the Navy	(904) 542-6826	See Worksheet #1 for signature	All	
Mike Pattison	Navy/ IRP Manager/ NAS Whiting Field POC	(850) 623-7268		All	
Craig Benedikt	USEPA/ RPM/ Provides Regulator Input	(404) 562-8555	See Worksheet #1 for signature	All	
John Winters	FDEP/ RPM/ Provides Regulator Input	(850) 245-8999	See Worksheet #1 for signature	All	

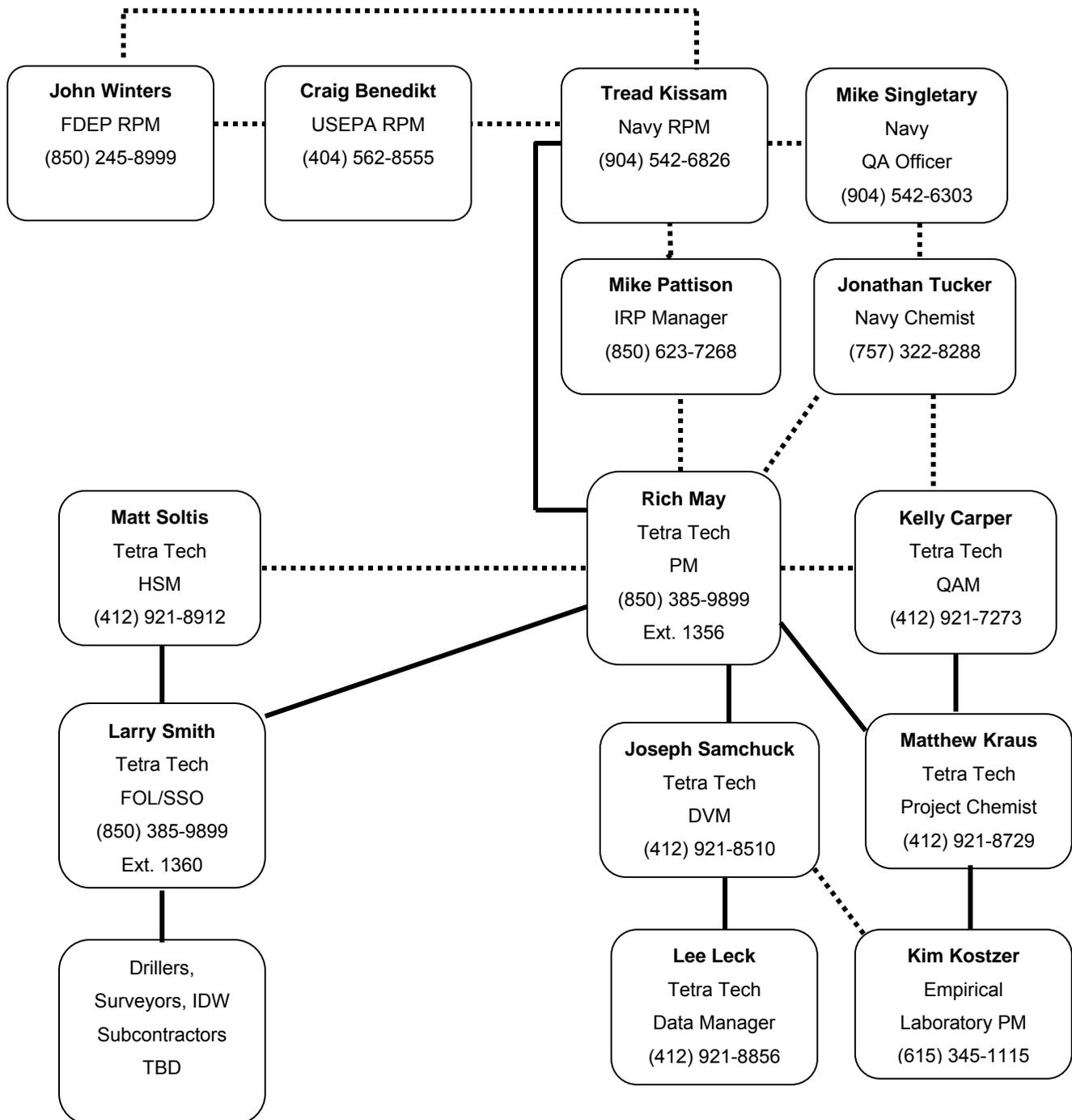
Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
<b>Tetra Tech Project Team Personnel</b>					
Rich May	Tetra Tech/ PM/ Manages Project Activities	(850) 385-9899 Ext. 1356	See Worksheet #1 for signature	All	
Larry Smith	Tetra Tech/ FOL and SSO/ Manages Field Operation and Site Safety Issues	(850) 385-9899 Ext. 1360		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	
Matthew Kraus	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory(s)	(412) 921-8729		All	
Joseph Samchuck	Tetra Tech/ DVM/ Manages Data Validation	(412) 921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	
Lee Leck	Tetra Tech/ Data Manager/ Manages Databases	(412) 921-8856		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
<b>Subcontractor Personnel</b>					
Kim Kostzer	Empirical/ Laboratory PM/ Representative for Laboratory and Analytical Issues	(615) 345-1115		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-#36	
Driller (TBD)	TBD, Subcontractor PM/Driller for DPT and Monitoring Well Installation	TBD		Worksheets #6, #14, #17, and Figures	
Surveyor (TBD)	TBD, Surveyor/Provides Surveying Services	TBD		Worksheet #14 and Figures	
IDW Subcontractor (TBD)	TBD, IDW Subcontractor/Provides IDW Services	TBD		Worksheet #14	

**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	Larry Smith Rich May Tread Kissam	(850) 385-9899 Ext. 1360 (850) 385-9899 Ext. 1356 (904) 542-6826	<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>
Changes in schedule	Tetra Tech PM Navy RPM NAS Whiting Field IRP Manager	Rich May Tread Kissam Mike Pattison	(850) 385-9899 Ext. 1356 (904) 542-6826 (850) 623-7268	The Tetra Tech PM will verbally inform the Navy RPM and the 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
Issues in the field that result in changes in scope of field work	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM NAS Whiting Field IRP Manager	Larry Smith Rich May Tread Kissam Mike Pattison	(850) 385-9899 Ext. 1360 (850) 385-9899 Ext. 1356 (904) 542-6826 (850) 623-7268	<p>The Tetra Tech FOL will verbally inform the Tetra Tech PM on the day that the issue is discovered.</p> <p>The Tetra Tech PM will inform the Navy RPM and the IRP Manager (verbally or via e-mail) within one business day of discovery.</p> <p>The Navy RPM will issue scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed.</p> <p>The Tetra Tech PM will document the change via an FTMR form within two days of identifying the need for change and will obtain required approvals within five days of initiating the form.</p>
Recommendation to stop work and initiate work upon corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Tetra Tech HSM Navy RPM NAS Whiting Field IRP Manager	Larry Smith Rich May Kelly Carper Matt Soltis Tread Kissam Mike Pattison	(850) 385-9899 Ext. 1360 (850) 385-9899 Ext. 1356 (412) 921-7273 (412) 921-8912 (904) 542-6826 (850) 623-7268	<p>If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform on-site personnel, subcontractor(s), the 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>

<b>Communication Drivers</b>	<b>Responsible Affiliation</b>	<b>Name</b>	<b>Phone Number and/or E-Mail</b>	<b>Procedure</b>
Corrective action for field program	Tetra Tech QAM Tetra Tech PM	Kelly Carper Rich May	(412) 921-7273 (850) 385-9899 Ext. 1356	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.  The Tetra Tech PM will then notify the Navy RPM within one business day.
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech PM	Larry Smith Rich May	(850) 385-9899 Ext. 1360 (850) 385-9899 Ext. 1356	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.
Analytical data quality issues	Empirical Laboratory PM Tetra Tech Project Chemist	Kim Kostzer Matt Kraus	(615) 345-1115 (412) 921-8729	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered.  The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation staff and the Tetra Tech 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
Tread Kissam	Navy RPM/ Manages Project Activities for the Navy	NAVFAC SE	Oversees project implementation, including scoping, data review, and evaluation.
Mike Pattison	IRP Manager/ Manages daily site activities related to this project	NAS Whiting Field	Oversees site activities, participate in scoping, data review, and evaluation.
Craig Benedikt	RPM/ Provides Regulator Input	USEPA Region 4	Participates in scoping, data review, evaluation, and approves the SAP.
John Winters	RPM/ Provides Regulator Input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP.
Rich May	PM/ Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day to day management of the project.
Larry Smith	FOL and SSO/ Manages Field Operation and Site Safety Issues	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities.
Kelly Carper	QAM/ Oversees program and project QA activities	Tetra Tech	Ensures that quality aspects of the CLEAN Program are implemented.

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.
Matt Kraus	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 quality assurance 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>• Ensure quality assurance of data validation deliverables.</li> <li>• Provide technical advice on data usability.</li> <li>• Coordinate and maintain data validation review schedule.</li> </ul>
Lee Leck	Data Manager/ Oversees database activities	Tetra Tech	Manages Tetra Tech databases and ensures input of data.
Kim Kostzer	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
TBD	Driller	TBD	Performs Rotasonic, Direct Push Technology (DPT) and/or Push-Ahead™ soil borings according to scope of work. Installs permanent monitoring wells.
TBD	Surveyor	TBD	Determines location data for soil boring and well locations according to scope of work.
TBD	IDW Subcontractor	TBD	Responsible for transport and disposal of IDW according to scope of work.

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

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

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

Project Name: NAS Whiting Field Projected Date(s) of Sampling: 26 August 2009 Project Manager: Rich May		Site Name: Site 40, Basewide Groundwater  Site Location: Milton, Florida			
<b>Date of Session:</b> August 26, 2009					
<b>Scoping Session Purpose:</b> Develop DQOs					
Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Craig Benedikt	RPM	USEPA Region 4	(404) 562-8555	benedikt.craig@epamail.epa.gov	RPM
Rich May	PM	Tetra Tech	(850) 385-9899 Ext. 1356	rich.may@tetrattech.com	PM
Mike Jaynes	Timekeeper	Tetra Tech	(850) 385-9899 Ext. 1354	mike.jaynes@tetrattech.com	Engineer
Mike Pattison	POC	NAS Whiting Field	(850) 623-7268	michael.pattison@navy.mil	POC
Larry Smith	Leader	Tetra Tech	(850) 385-9899 Ext. 1360	larry.smith@tetrattech.com	FOL
Amy Twitty	Guest	CH2MHill	(850) 939-8300	atwitty@ch2m.com	Geologist
John Winters	State RPM	FDEP	(850) 245-8999	john.winters@dep.state.fl.us	RPM
Gus Campana	Facilitator	Campana Consulting	(407) 352-3687	gusbell@earthlink.net	Facilitator
Jacqueline Strobl	Scribe	Tetra Tech	(850) 385-9899 Ext. 1351	jacqueline.strobl@tetrattech.com	Scribe
Mike Singletary	Senior Technical	NAVFAC SE	(904) 542-6303	michael.a.singletary@navy.mil	QA Officer
Mike Maughon	Senior Technical	Tetra Tech	(843) 886-4547	mike.maughon@tetrattech.com	Consultant
Peggy Churchill	DQO Facilitator	Tetra Tech	(321) 636-6470	peggy.churchill@tetrattech.com	DQO Facilitator
Katie Newman	Project Scientist	Tetra Tech	(850) 385-9899 Ext. 1357	katie.newman@tetrattech.com	Project Scientist
Elliot Jones	Hydrologist	U.S. Geological Survey	(404) 562-8505	<a href="mailto:jones.elliott@epa.gov">jones.elliott@epa.gov</a>	Hydrologist

### **Comments/Decisions:**

The meeting held on August 26, 2009 was to provide all parties the opportunity to have a “buy in” and come to an agreement on how to proceed forward. The DQOs were addressed in three steps.

Step 1: State the Problem. During this step, the Site history was reviewed; DQO Facilitation process was begun; receptors at Site 40 were discussed; data required to complete the RI was identified; and data gaps from previous event were discussed.

Step 2 (combined with Step 5): Study Goals and the Analytic Approach. During this step, items discussed included collecting data to delineate the North Field plume. The team also reviewed, discussed, and adjusted the proposed well locations for the trichloroethene (TCE) plume based on each team member’s input, suggestions, and concerns.

Step 3: Decision Inputs. Information inputs discussed are as follows: Data from nested wells installed during Site 4 investigation; moving monitoring well WHF-1467-MW-40S to the north and WHF-1467-MW-43S to the north; Chemical data TCE and benzene, toluene, ethylbenzene, and total xylenes (BTEX) from newly installed wells; Groundwater field parameters; and previously collected data.

### **Action Items:**

- Action Item #082609-01: Tread Kissam- Provide the monitoring data from the Basic Ordering Agreement contractor to the team.
- Action Item #082609-02: Tread Kissam - Review FDEP comments on the Site Assessment Report (SAR) for Site 2894 to determine if new sampling needs to occur or if a new well needs to be installed.
- Action Item #082609-03: Tetra Tech (Larry Smith, Rich May, and Mike Jaynes) – Review the data for Site 2894.

### **Consensus Decisions:**

- Consensus #082609-01: Source area data gaps will be addressed in the Site 40 FS.

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

**10.1 INTRODUCTION**

Site 40 - Basewide Groundwater, also known as OU 25, encompasses the entire geographic area of the base. The primary focus of this SAP is to address data gaps in the commingled BTEX and TCE plumes located in the North Field Industrial Area discovered when several wells were determined to be dry. A secondary priority is to collect supplemental current data at a separate BTEX plume associated with UST Site 1438/1439 and to investigate UST Site 2894. The resultant data will be used to complete the RI Report Addendum for Site 40 Basewide Groundwater. Information related to site location, description, history, contaminant sources, potential contamination routes, and possible exposure pathways to human receptors was used to develop the Site 40 Conceptual Site Model (CSM) described below. The CSM serves as the basis for developing this UFP-SAP.

**10.2 SITE AND LOCATION DESCRIPTION**

Whiting Field is located in Santa Rosa County, in Florida's northwest coastal area, 5.5 miles north of Milton and 25 miles northeast of Pensacola (Figure ES-1). NAS Whiting Field is approximately 3,842 acres in size, and consists of two airfields (North Field and South Field) each with associated hangars and industrial areas for support activities. NAS Whiting Field, home of Training Air Wing Five, was constructed in the early 1940s. It was commissioned in July 1943 and has served as a naval aviation training facility since then. The field's mission is to train student naval aviators in fixed-wing propeller-driven aircraft, and basic and advanced helicopter training. The North Field is used for fixed-wing aircraft training, while the South Field is used for helicopter training.

Land surrounding NAS Whiting Field consists primarily of agricultural land to the northwest, residential and forested areas to the south and southwest, and forests along the remaining boundaries. The facility is bounded on the west by Clear Creek, a tributary to the Blackwater River which discharges to the estuarine waters of the East Bay of the Escambia Bay coastal system.

Elevation at NAS Whiting Field is about 190 feet above mean sea level (msl) with a drop in elevation of approximately 150 feet to Clear Creek at 45 feet above msl. NAS Whiting Field is drained by an extensive storm sewer drainage system constructed in the mid-1940s. Extensive slope contouring and paved drainage ditches channel water from NAS Whiting Field to Clear Creek and to Big Coldwater Creek.

### **10.3 SITE HISTORY**

A variety of wastes related to pilot training, the operation and maintenance of aircraft and ground support equipment, and the station's facility maintenance activities have been generated at NAS Whiting Field throughout its years of operation. Prior to the establishment of hazardous waste management programs and programs to recycle waste oil, most of the hazardous wastes were reportedly disposed on-site in landfills typically located around the perimeter of the facility and at locations near tank farms.

In 1985, an Initial Assessment Study (Envirodyne Engineers, 1985) identified 15 sites for study. Additional sites were added to the list in the early 1990s and investigation of groundwater contamination for individual IRP and UST sites was underway. During this investigative period, two commingled BTEX and TCE plumes were located beneath NAS Whiting Field's North and South Field Industrial Areas.

Due to the presence of TCE in groundwater above USEPA maximum contaminant levels (MCLs), NAS Whiting Field was listed on the National Priorities List (NPL) in 1994 and has since been regulated under CERCLA.

In an effort to reduce the complexities dealing with commingled plumes, in 1997 the Project Team designated Site 40- Basewide Groundwater, as a site separate from the overlying contaminated soil. As a result, soil at all IR sites is addressed separately from groundwater. However, leachate from all IR and UST site soils must be addressed under Site 40.

In 1996, 1997, 1998, and 2000, groundwater contamination data sets were generated from investigations designed to assess small areas of interest, but these investigations did not utilize all facility monitoring wells (over 200). Due to the divided nature of these separate site investigations, a non-synoptic data set was assembled to represent a base-wide understanding of groundwater contamination by using the most recent data, primarily 1998 and 2000 data, for the 2003 RI Report (Tetra Tech, 2003). The Site 40 RI was submitted to and rejected by FDEP and the USEPA, not because of fault with the report, but rather the regulators determined that multiple IR sites with unresolved soil contamination issues overlying Site 40 could be leaching to groundwater. This variable could not be addressed by groundwater data alone. The regulators required signed Record of Decision (RODs) for soils on all IR sites indicating they were not leaching to Site 40. So, the focus shifted; during the next seven years, RODs were issued for soils on 18 facility sites (Sites 3, 5A, 6, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 30, 32, 33, 35, and 38). Currently, only Site 39, Clear Creek, located topographically and hydraulically downgradient of Site 40, Site 41 (which has a ROD pending in 2010), and Site 40 do not have signed RODs.

In 2007, the Partnering Team determined the groundwater analytical data set required supplemental data prior to revising and resubmitting the Site 40 RI Report, issued previously in April 2003. Because Site 40

is defined as Basewide Groundwater, this includes all wells associated with the two commingled plumes at North Field and South Field and the unresolved UST Site 1438/1439. Three basewide groundwater sampling events were planned with the expectation that data collected over the three years would show decreasing contaminant concentration trends.

However, a data gap became apparent in 2008 when 20 shallow wells on the eastern side of the North Field plume were found to be perched above the water table. A subsequent review of rainfall data revealed a regional drought began in approximately 2000 and continued through 2008. Due to the drought, the water table in the North Field area dropped an average of 8 feet. As a result, 20 shallow monitoring wells no longer extended to the surficial water table, but were perched above the water table and were dry, or provided perched water that did not reflect conditions of the surficial aquifer. These wells were primarily located on the eastern boundary of the North Field plume and include the wells at UST Site 2894.

Currently, UST Sites 4, 7, 1438/1439, and 2894 are acknowledged to be leaching to, or otherwise impacting Site 40. Site 4 is in the South Field area. Site 7 overlies and is the source for the BTEX component of the North Field commingled plume. Site 1438/1439 is a closed petroleum storage and distribution facility with a significant dissolved BTEX plume that has not been sampled since 1998 and requires sampling to collect current groundwater data. Site 1438/1439 has an active soil vapor extraction (SVE) system installed. Site 2894 is an active petroleum storage and distribution facility initially investigated in 1993 resulting in a vadose zone only passive barometric pumping remedial system. In 2004 floating product was detected prompting a new investigation. A SAR was issued to FDEP in 2007 recommending installation of two new wells to assess the underlying groundwater.

As a result of these findings, the NAS Whiting Field Project Team identified the need to install seven new monitoring wells in the surficial aquifer to re-delineate the eastern area of the North Field plume, and two new monitoring wells in the surficial aquifer to delineate the UST Site 2894 plume. Ten existing monitoring wells will be sampled to assess the northern, eastern, and southern boundary areas of the commingled plume, including seven monitoring wells at UST Site 1438/1439 and three wells at UST Site 2894. The resulting data will be used to delineate the North Field Plume, and provide current groundwater analytical data for all subject sites as required by the regulators evaluating the RI where the data will be presented.

#### **10.4 CONCEPTUAL SITE MODEL**

The potential sources for the commingled North Field plume are BTEX derived from the Site 4 USTs and appurtenances and TCE from a nearby undefined source. The appurtenances consists of buried pipelines which conveyed bulk fuel from Site 1438/1439 and buried pipelines that conveyed fuel to the

fight line or ramp distribution points where aircraft are parked. Based on historical documents and conversations with facility personnel, the fuel ranged from aviation gasoline (AVGAS) with lead from 1950 through the 1980s followed by JP-4 and JP-5 during later years of turboprop operation. It is suspected that USTs at Site 4 were cleaned with TCE and the resultant sludge was disposed of in pits around the Site 4 area. As a result, media that could be contaminated are subsurface soil (which is addressed separately from Site 40) and groundwater.

A general geologic interpretation from the land surface to approximately 150 feet below ground surface (bgs) is presented in subsection 1.4.5 of the GIR (ABB-ES, 1998). Further detail for this interval is presented in Chapter Three of the RI/FS Technical Memorandum No. 2 Geologic Assessment (ABB-ES, 1995).

The shallow surficial fine sands, above 28 feet bgs, have a high percentage of clays and silts that form a layer that resists the movement of subsurface or vadose zone gases. Drilling through this layer produces pathways for enhanced subsurface gas movement that is very responsive to barometric pressure variations. Below the clayey silty upper layer are loose quartz sands (28 to 85 feet bgs) that form a subsurface reservoir for vadose zone gases. When the upper clayey silty fine sands (0 to 28 feet bgs) are breached, vadose zone gases migrate in or out due to pressure atmospheric pressure fluctuations. Impermeable silty or clay layers located at depth (approximately 85 to 105 feet bgs) near the surficial aquifer act as platforms or shallow basins where leaching fuels from the UST Sites described above have collected over time. After rainfall events, the basins fill with infiltrating rain water and product floating on this water is displaced and spills over the basin side, eventually impacting the surficial aquifer.

#### **10.4.1 Source Media, Migration Routes, and Release Mechanism**

The source media typically consist of contaminated subsurface soil from approximately 15 feet bgs to the water table at approximately 110 feet bgs at all sites under investigation. Petroleum contaminants are found at all sites under consideration. TCE is present in groundwater underlying Site 4 but a vadose zone source has not been located. At Sites 4 and 2894 free product has been detected in soil overlying low permeability clay lenses just above the water table. The remaining site has known vadose zone contamination with underlying groundwater contamination. Rain water infiltration is the likely transfer mechanism from vadose soil to groundwater.

There are two migration route end points for groundwater from the source area(s). The first is the facility water distribution system, which consists of three public water supply wells are located at NAS Whiting Field. These supply wells provide potable water for general use by base personnel, that includes drinking and bathing at all facility buildings such as the Combined Bachelors Quarters where pilot trainee's and their families board for the duration of their training. This local water supply has been treated by a

granular activated carbon (GAC) system maintained by the Navy and monitored on a monthly basis for BTEX and TCE since 1986. Monthly testing is conducted to ensure that water provided to base personnel meets FDEP and USEPA drinking water criteria.

The second end point occurs as upwelling groundwater enters Clear Creek and the adjoining floodplain. Potential exposures and human health and ecological risks at Clear Creek will be assessed in the Site 39 RI.

Due to the porosity of the soil, contaminants have reached groundwater. Other migration pathways could be soil to soil gas, soil gas to indoor air, and soil gas to ambient air. The release(s) from Site 4 likely occurred from the 1940s to the 1990s, and as a result, contaminants likely range from degraded AVGAS fuels used in the early operations at the facility to more recent formulations of JP-4 and JP-5. In 1998 and again in 2008, 18 inches of petroleum-based product was measured in monitoring well WHF-1467-MW-26P, which terminates on a perched clay layer located approximately 100 feet bgs, about 10 feet above the surficial water table. Subsequent product detections in nearby wells indicate a continuous perched product mass may measure approximately 50 by 150 feet.

The North Field BTEX plume originates near Site 4 and migrates southwest.(hydraulically downgradient) approximately 2,000 feet from the southeastern boundary of Site 4, where BTEX and TCE concentrations exceeding FDEP and USEPA criteria, drop to below 1 microgram per liter ( $\mu\text{g/L}$ ). BTEX and TCE (and/or daughter products) have been detected in groundwater samples collected from monitoring wells located between the toe of the plume and at Clear Creek 7,600 feet to the southwest. "Seepage velocities (or average linear velocities) ranged from 0.11 to 1.38 feet/day" (ABB-ES, 1995). These average linear velocities indicate BTEX and TCE traveled from the source area to Clear Creek in approximately 30 years. If Site 4 contaminants reached the groundwater in the 1950s, Clear Creek could have been impacted as early as the 1980s.

Acetone, benzene, 2-butanone, 1,2-dichloroethene (total), TCE, toluene and vinyl chloride (VC) have been detected upwelling into the Clear Creek floodplain at concentrations that exceed FDEP Groundwater Cleanup Target Levels (GCTLs) and USEPA primary drinking water standards. Additionally, 1,1-dichloroethene, 1,2-dichloroethene (total), benzene, TCE, and VC have been detected in groundwater beneath the Clear Creek stream bed. Benzene, TCE, and VC were detected at concentrations that exceed the FDEP GCTLs and the USEPA primary drinking water standards. These contaminants migrate from the two commingled plumes located beneath the North and South Field Industrial Areas of the facility and then migrate to the southwest and upwell into Clear Creek, where the low concentrations are rapidly diluted and become non-detectable in Clear Creek surface water samples.

Exposure pathways in the Clear Creek area to human and ecological receptors will be addressed in the Site 39 RI.

At the request of the USEPA and FDEP groundwater from all new wells are analyzed for metals. The resultant metals analytical data is used to confirm metals are not impacting the aquifer and to support evaluation of natural attenuation processes.

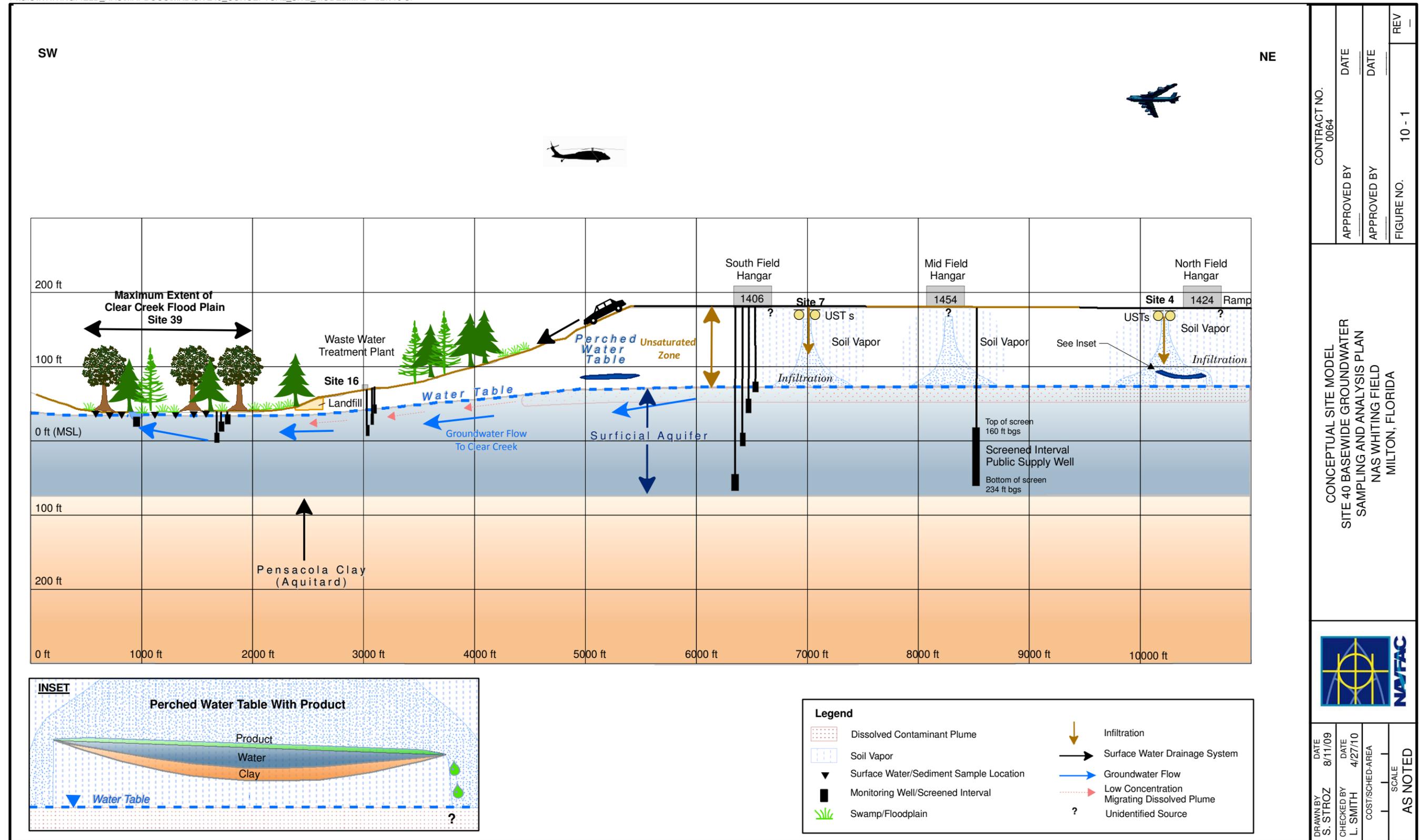
#### **10.4.2 Land Use, Potential Receptors, and Exposure Pathways**

The current land use patterns at the site are well established, thereby reducing the uncertainty associated with land use assumptions. Access to NAS Whiting Field is restricted to military personnel, civilian employees, and authorized visitors. The installation is surrounded by a perimeter fence. Signs posted on the fence warn that trespassing is not permitted. People entering the facility must pass through staffed entrance gates. Within installation boundaries, certain contaminated sites are fenced. The use of facility operated public supply wells providing on-site treated water will continue. The current land use and future use will be the same.

The two receptors are on-facility residents and construction workers. Residential receptors are assumed to use groundwater for domestic purposes (i.e., bathing, showering, washing dishes), which may result in dermal exposure and inhalation of volatiles if the GAC were not in place. Ingestion of groundwater may result in the intake of groundwater contaminants of potential concern (COPCs). It is also possible, under future land use conditions where excavations are present at NAS Whiting Field, for construction workers and site occupational workers to be dermally exposed to contaminated soil. For construction workers and on-site occupational workers, exposure to constituents via inhalation is expected to be minimal in areas outside of the boundaries of Site 4. However, reports of strong fumes have also been reported in areas around Site 32, the North Field Hangar. The CSM is shown on Figure 10-1.

At Site 40, there is no complete exposure pathway for ecological receptors. Exposure of ecological receptors to groundwater that upwells into Clear Creek will be addressed in the Site 39 RI.

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

**11.1 PROBLEM STATEMENT**

Previous Site 40 groundwater investigations have described the western extent of the North Field TCE and BTEX commingled plume. Water levels measured in 2008 showed that 20 monitoring wells located on the eastern side of the plume were not installed to a depth that reaches the surficial water table. All previous groundwater analytical data from these wells is now known to reflect conditions in a perched aquifer instead of the surficial sand-and-gravel aquifer. Therefore, the eastern boundary of the North Field plume within the surficial aquifer must be redefined. The collected data will be used to supplement the Site 40 RI Report, which will be revised and resubmitted based on the additional data from the monitoring wells that are sampled as part of this investigation. A secondary issue requires sampling of monitoring wells at UST Sites 2894 and 1438/1439, in order to update the CSM and present the current conditions of groundwater at these sites in order to complete the Site 40 RI Report.

**11.2 IDENTIFY INFORMATION INPUTS**

Data will be collected at specific areas of Site 40 for purposes of identifying the horizontal and vertical extent of contamination from COPCs associated with the UST and IRP sites and for updating the CSM. In order to meet the study goals of the investigation, the physical and chemical data to be collected at Site 40 are described below:

1. Previously collected data: Data that was presented in the 2003 Site 40 RI Report will be used with the newly collected data for a presentation of the current CSM in the revised Site 40 RI Report.
2. Groundwater Field Investigation Parameters: Groundwater measurements for elevation, dissolved oxygen (DO), conductivity, pH, temperature, turbidity, and oxidation-reduction potential (ORP) parameters will be collected. Standard field screening Standard Operating Procedures (SOPs) (identified in Worksheet #21) will be used for collecting these data.
3. Chemical Analysis: Groundwater analytical data from newly installed and existing monitoring wells will be added to the existing Site 40 data set to determine the extent of one or more of the target analytes presented in Worksheet #15. The sampling methods are presented in Worksheet #18 and the analytical methods are presented in Worksheet #19. The selected target analytes represent those analytes that are associated with groundwater contamination at Site 40 and the CSM.

4. Site Survey: A site survey will be conducted for all wells installed since 2000. The information collected will be added to the Tetra Tech Geographic Information System (GIS) database.
5. Project Action Limits (PALs): This investigation requires analytical chemical data that can be used to delineate groundwater contamination through comparison to conservative screening values (i.e., PALs). For this investigation, the PALs are listed and described below:
  - FDEP GCTLs - Residential and Industrial Use per Chapters 62-770 and 62-777, Florida Administrative Code (F.A.C.).
  - USEPA Regions 3, 6, and 9, Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites, December 2009 – Tap Water.

In cases where the PAL is between the laboratory limit of quantitation (LOQ) and the method detection limit (MDL), FDEP will analytical results below the LOQ for that analyte as long the validated results are “J” qualified. In cases where the PAL is less than the MDL, the Project Team has agreed to replace the PAL with the laboratory LOQ for decision-making purposes, as is suggested in “Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits” (FDEP, 2004). The Partnering Team agrees that if some chemicals are not detectable at levels commensurate with PALs, these analytes and their results will be will be addressed in the risk assessment uncertainty analysis in the revised Site 40 RI Report.

It will be necessary to collect sufficient sample media to support quality control (QC) activities. Field and laboratory QC requirements are presented in Worksheets #12 and #20.

### **11.3 STUDY BOUNDARIES**

The following items address the horizontal and vertical boundaries for the Site 40 investigation. The aim of this investigation is to establish the North Field plume’s northern, eastern, and southern boundary utilizing data from new monitoring wells installed into the surficial aquifer and new data from specific existing monitoring wells and to provide new data for certain wells where data gaps were identified. The horizontal and vertical boundaries of UST Site 1438/1439 are predetermined, only previously installed wells will be sampled.

#### North Field Plume Horizontal Boundary:

The North Field plume is spatially bounded by monitoring well cluster WHF-32-series on the west side, is currently unbound on the east side, north ramp on the north side, and is currently unbound on the south side. The North Field plume is approximately 2,700 feet by 1,100 feet (Figure 17-1). This defines the horizontal boundary for the investigation.

#### North Field Plume Vertical Boundary:

The vertical groundwater investigation boundary is defined by the current potentiometric surface of the surficial water table found at an average depth of approximately 110 feet bgs. Dissolved BTEX compounds have a specific gravity of less than 1.0 and are typically found in the upper surficial groundwater, so this is the target depth. Replacement well screens will be 20 feet in length and designed to straddle the water table with 5 feet of screen in the vadose zone. This defines the vertical boundary for the investigation.

#### Temporal Boundary:

Regarding temporal boundaries, groundwater conditions are not expected to change significantly over a 6-month to 1-year time frame. Any particular season or seasons have not been identified as most optimal for sampling and tidal influence is not a concern.

Also, water level measurements will be collected within 24 hours of the initiation of the sampling event to ensure that the measurements are representative of the same time frame. These measurements should also not be made sooner than 24-hours after a significant precipitation event, as decided by the Tetra Tech PM.

One population of interest is shallow groundwater located along the northern, eastern, and southern perimeter, or hydrogeologically upgradient, side gradient, or downgradient within the surficial aquifer of the North Field plume (Figure 17-1). Replacement wells are being positioned to provide groundwater samples from plume margins or just beyond the margin in non-contaminated areas to aid in determination of the plume contamination gradient. Other populations of interest include shallow surficial aquifer groundwater at UST Sites 2894 and 1438/1439. Representative samples from the populations of interest will be collected and used for decision making.

### **11.4 ANALYTIC APPROACH**

This SAP was developed to address data gaps identified during the 2008 water level investigation. The objective of groundwater monitoring at Site 40 is to delineate the extent of contamination from target analytes including BTEX and TCE at source areas that have not been completely defined.

One round of groundwater samples will be collected from new and existing monitoring wells and analyzed for one or more of the target analytes presented in Worksheet #15. Based on the information identified in Worksheet #10, a decision rule was developed to govern data use. The decision rule includes the direct comparison of the laboratory analytical results to the applicable PAL to determine if there is an exceedance of any applicable regulatory criteria.

Collect data to delineate the extent of target analyte contamination at the North Field plume and UST Sites 1438/1439 and 2894. If any analytical result is greater than the PAL for any target analyte in the perimeter wells designated for the plume associated with that site, then delineation is not complete and a recommendation for further data collection will be made in the revised Site 40 RI Report. If all analytical results within the sampled wells are less than the PAL for the target analytes, then delineation is complete and the data will be presented in the revised Site 40 RI Report.

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

The intent is to efficiently delineate the extent of contamination in the areas of interest while achieving wide spatial coverage to fill data gaps. Because the sampling design is judgmental, and has been agreed upon by the Partnering Team, no statistical computation of the required number of samples and no specification of statistically based decision performance objectives shall be necessary. Simple comparisons of measured concentrations to action levels are being used. The project team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have action levels which are less than the 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. All stakeholders will be involved in rendering the final conclusion regarding adequacy of the data.

#### **11.6 DEVELOP THE DETAILED PLAN FOR OBTAINING DATA**

The sampling plan and rationale for the collection of data to fill in data gaps at Site 40 is included in Worksheet #17. Figure 17-1 represents the proposed sample locations.

**SAP Worksheet #12 -- Measurement Performance Criteria Table - Field Quality Control 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)
Field Blank	All Fractions	One per source water.	Accuracy/Bias/Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment <sup>1</sup> .	Accuracy/Bias/Contamination	No analytes $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be < LOQ.	S&A
Trip Blanks	Volatile Organic Compounds (VOCs) (BTEX and TCE+)	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 Duplicate	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

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.

TCE+ = Trichloroethene, tetrachloroethene, plus daughter products: cis-1,2-dichloroethene, trans-1,2-dichloroethene, and vinyl chloride

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

<b>Secondary Data</b>	<b>Data Source</b> (originating organization, report title and date)	<b>Data Generator(s)</b> (originating organization, data types, data generation / collection dates)	<b>How Data Will Be Used</b>	<b>Limitations on Data Use</b>
Information Report	<i>NAS Whiting Field General Information Report</i> 1998	ABB Environmental Services/ Remedial Investigation/Feasibility Study, 1974	Previous site information	None
Remedial Investigation	<i>Site 40 Basewide Groundwater Remedial Investigation Report (Draft)</i>	Tetra Tech NUS, Inc./ Remedial Investigation/ 1998-2000	Previous investigation data	None

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

**14.1 SUMMARY OF PROJECT TASKS**

Field activities include the following tasks:

- Mobilization/Demobilization (including site-specific health and safety training)
- Roto-Sonic Boring
- Monitoring Well Installation
- Monitoring Well Elevation Surveying
- Groundwater Sampling
- Water Level Measurements
- Field Instrument Calibration
- Field Decontamination Procedures
- IDW Management

Additional project activities include the following tasks:

- Documentation and Records
- Data Packages
- Data Review Tasks and Third Party Data Validation
- Analytical Tasks
- Data Management Tasks
- Data Assessment and Oversight Tasks
- Data Review Tasks
- Project Reports

**14.1.1 Mobilization/Demobilization**

Following approval of this SAP, Tetra Tech will procure the required field subcontractors and begin mobilization activities. Mobilization/demobilization will include the following:

- Completion of utility clearances in the proposed boring areas (receipt of dig permits).
- Mobilization of equipment and materials to the site.
- Receipt of drilling and/or dig permits.
- Meet with NAS Whiting Field Air Operations to define area of work relative to aircraft parking and flight operations and obtain flight line driving permits if necessary.

- Conductance of a site-specific health and safety review meeting.
- Delineation of the work zones (exclusion zone, contamination reduction zone, and support zone) as required by the HASP.
- Arrangement of an area to perform decontamination procedures.
- Demobilization of equipment and materials from the site.
- Performance of general site cleanup.
- Field team members will review this SAP and the associated project HASP. Mobilization includes attendance at a site-specific health and safety kick-off meeting during the initiation of on-site activities. This meeting will also include field team orientation in order to familiarize personnel with the scope of the field activities and obtain ramp and runway drivers licenses.

The Tetra Tech FOL will coordinate the mobilization activities. The Tetra Tech FOL responsibilities include initiating and conducting equipment inventories to ensure equipment is available, purchasing equipment as required, staging equipment for efficient loading and transport from the Tetra Tech office to the site, and, after field activities are completed, demobilizing the equipment.

The Tetra Tech FOL will coordinate with the Navy on-site contact regarding passes, security and access issues, and daily activities. The Tetra Tech FOL will also coordinate with the Navy RPM and stakeholders regarding field activities. Demobilization will include transporting personnel, field equipment, supplies, and the drilling subcontractor from the site, performing general site cleanup, and organizing and finalizing field paperwork.

#### **14.1.2 Monitoring Well Installation**

Monitoring wells will be installed using Roto-Sonic technology at or near the locations identified on Figure 17-1. Wells will be installed in accordance with Tetra Tech SOP GH-1.3 and FDEP SOP PCS-006 (Appendix A). Nine new monitoring wells will be installed at Site 40. These wells are intended to monitor contaminant concentrations in shallow surficial groundwater. The shallow surficial wells will be installed to an approximate depth of 120 feet bgs with a 20-foot well screen bracketing the water table. The actual depth of the screened interval will be based on field observations of the lithology at each location.

The monitoring wells will be installed and constructed in accordance with NAVFAC SE and FDEP guidance documents. The monitoring wells will be of a 2-inch nominal diameter, and will be constructed of Schedule 40 polyvinyl chloride (PVC). The monitoring wells installed during this phase will be developed through over pumping after installation, but not before a 24-hours set time from the final annular grouting.

### **14.1.3 Monitoring Well Elevation Surveying**

The horizontal location, elevation, and top-of-riser elevations for each of the new or replacement monitoring wells and other wells that have been installed at Site 40 since 2000 will be surveyed by a Florida registered professional land surveyor. Top-of-riser elevations will be surveyed to the nearest 0.01 foot and will be tied into the existing site datum (North American Vertical Datum 1988).

### **14.1.4 Groundwater Sampling**

Groundwater samples will be collected from the newly installed monitoring wells after a minimum of 24 hours has passed after well development. Samples will be collected from the nine newly installed monitoring wells and additional samples will be collected from 10 existing monitoring wells. Groundwater samples 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. Samples will be collected in accordance with Tetra Tech SOP SA-1.1 and FDEP SOPs FS1000 and FS2200 (Appendix A).

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 aliquot for VOC 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.

### **14.1.5 Water Level Measurements**

One synoptic round of water-level measurements will be conducted at each monitoring well that will be sampled to provide information regarding groundwater flow patterns and 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.

#### **14.1.6 Field Instrument Calibration**

The instruments used in the field will be calibrated as it states in the manufacturer equipment manual. The equipment that will require calibration are provided in Worksheet #22. Calibration will be documented on the Equipment Calibration Log (Appendix B).

#### **14.1.7 Field Equipment Decontamination**

Sample containers will be provided certified-clean from Empirical. Sampling equipment will be decontaminated prior to and between sampling at each location. The decontamination procedures contained in Tetra Tech SOP SA-7.1 (Appendix A) will be followed for this project.

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

Disposable sampling equipment, used personal protective equipment (PPE), and general project refuse will be collected and placed in plastic bags (double-lined) and disposed in a NAS Whiting Field municipal trash receptacle. Waste water and soil generated during field activities will be containerized and sampled for proper disposal characterization. Tetra Tech will be conducting the characterization and will send the results to a subcontractor who will dispose of the IDW properly. The Facility is the generator and will sign the waste manifest.

#### **14.1.9 Documentation and Records**

Documentation of sample location coordinates, borings logs, chain-of-custody forms, samples logs, and shipping documents for all samples will be recorded and kept. Also, electronic and hardcopies of this SAP will be kept on-site and in the Tetra Tech project files.

#### **14.1.10 Data Packages**

Data packages will include receipt of analytical data packages from Empirical and generation of Tetra Tech data validation reports.

#### **14.1.11 Data Review Tasks and Third Party Data Validation**

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

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

## **14.2 ANALYTICAL TASKS**

Chemical analyses for VOCs (BTEX and TCE+) and metals will be performed by Empirical, a subcontracted laboratory located in Nashville, Tennessee. Empirical holds current accreditation through the DoD Environmental Laboratory Accreditation Program (ELAP). Analyses will be performed in accordance with the analytical methods identified in Worksheet #30 and the requirements of the technical specification for laboratory services developed by Tetra Tech for this SAP. Empirical will meet the LOQs specified in Worksheet #15.

The technical specification details the analytical requirements, number of samples, matrix, analytical methods to be performed, preservatives, holding times, the LOQs required for the project, and data deliverables. Empirical will perform the chemical analyses following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheets #19 and #30. Copies of the laboratory SOPs are included in Appendix C.

## **14.3 DATA MANAGEMENT TASKS**

Data management activities are crucial to ensure reliability and maintain organization of the site data. This section describes essential data management activities which include: data handling, data tracking and control, and record keeping.

- Project documentation and records
  - Field sample collection and field measurement records are described in Worksheets #27 and #29.
  - Laboratory data package deliverables are described in the analytical specifications.
  - Data assessment documents and records are listed in Worksheet #29.
- Data recording formats are described in Worksheet #27.

### **14.3.1 Data Handling**

After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field 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. Corrections to entries made in field and laboratory logs will be made by striking through the erroneous entry with a single line and entering the correction nearby, with the date of correction and initials of the person making the correction.

#### **14.3.2 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.** A “cradle to grave” sample tracking system will be used from the beginning to the end of the investigation to track and control the data generated during the investigation. The Tetra Tech FOL will initiate the sample tracking process by ensuring that sample jar labels are complete and adhere to SAP requirements. When field sampling is underway, the Tetra Tech FOL will forward the chain-of-custody forms to the Tetra Tech PM via scanning and attaching to an email at the end of the day. The Tetra Tech PM or designee will compare the entries on the chain-of-custody forms with the SAP to confirm that the correct information is being collected/ requested. This will allow for early detection of errors made in the field. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contract laboratory. Upon receipt of the data packages from Empirical, 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 Empirical.
- **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field log books, 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 subject to audit to verify 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.

### **14.3.3 Record Keeping**

A number of documents must be completed before, during, and after the sampling event. These documents include at a minimum: chain-of-custody sheets, field data sheets, field books, field notes, photographs, and analytical data. In addition, adherence to sample holding times, sample preservation, and container requirements must also be documented. Field and analytical documentation will be maintained in the Tetra Tech project database and project file, as presented in Worksheet #29.

## **14.4 DATA ASSESSMENT AND OVERSIGHT TASKS**

Refer to Worksheet #31 for planned project assessments, Worksheet #32 for assessment findings and corrective actions, and Worksheet #33 for QA management reports.

## **14.5 DATA REVIEW TASKS**

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

## **14.6 PROJECT REPORTING**

Data collected as documented in this SAP will be prepared documenting the sampling activities and results of the data gaps investigation at Site 40 and combined with data reported previously in the April 2003 RI.

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

Matrix: Groundwater

Analytical Group: Metals

Analyte	CAS Number	Project Action Limit <sup>(1)</sup> (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical	
					LOQ (ug/L)	MDL (ug/L)
ALUMINUM	7429-90-5	200	FDEP Residential GCTL	67	200	50
ANTIMONY	7440-36-0	6	FDEP Residential GCTL	2.0	4 <sup>(2)</sup>	1.3 <sup>(2)</sup>
ARSENIC	7440-38-2	10	FDEP Primary standard	3.3	1.3	0.75
BARIUM	7440-39-3	2,000	FDEP Residential GCTL	670	40	10
BERYLLIUM	7440-41-7	4	FDEP Residential GCTL	1.3	1.3	0.50
CADMIUM	7440-43-9	5	FDEP Residential GCTL	1.7	1.3	0.50
CALCIUM	7440-70-2	NA	FDEP Residential GCTL	NA	5,000	1,000
CHROMIUM	7440-47-3	100	FDEP Residential GCTL	33	10	5
COBALT	7440-48-4	140	FDEP Residential GCTL	47	20	10
COPPER	7440-50-8	1,000	FDEP Residential GCTL	330	25	10
IRON	7439-89-6	300	FDEP Residential GCTL	100	100	50
LEAD	7439-92-1	15	FDEP Residential GCTL	5.0	5	2.5
MAGNESIUM	7439-95-4	NA	FDEP Residential GCTL	NA	5,000	1,000
MANGANESE	7439-96-5	50	FDEP Residential GCTL	17	15	5
MERCURY	7439-97-6	2	FDEP Residential GCTL	0.67	0.2	0.1
NICKEL	7440-02-0	100	FDEP Residential GCTL	33	10	5
POTASSIUM	7440-09-7	NA	FDEP Residential GCTL	NA	5,000	1,000
SELENIUM	7782-49-2	50	FDEP Residential GCTL	17	10	5
SILVER	7440-22-4	100	FDEP Residential GCTL	33	10	5
SODIUM	7440-23-5	160,000	FDEP Residential GCTL	53,000	5,000	1,000
THALLIUM	7440-28-0	2	FDEP Primary standard	0.67	2 <sup>(2)</sup>	0.75 <sup>(2)</sup>
VANADIUM	7440-62-2	49	FDEP Residential GCTL	16	15	5
ZINC	7440-66-6	5,000	FDEP Residential GCTL	1,700	20	10

(1) The Project Action Limit is the Florida Administrative Code Chapter 62-777 Groundwater Cleanup Target Level (GCTL) for groundwater samples. April 2005.

(2) Concentrated 4X per USEPA 200.7.

**Matrix: Groundwater**

**Analytical Group: VOCs (BTEX and TCE+)**

Analyte	CAS Number	Project Action Limit <sup>(1)</sup> (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical	
					LOQ (ug/L)	MDL (ug/L)
<b>BTEX Analytes</b>						
BENZENE	71-43-2	1	FDEP Residential GCTL	0.33	1	0.3
TOLUENE	108-88-3	40	FDEP Residential GCTL	13	1	0.3
ETHYLBENZENE	100-41-4	30	FDEP Residential GCTL	10	1	0.3
TOTAL XYLENES	1330-20-7	20	FDEP Residential GCTL	6.7	1	0.3
<b>TCE+ Analytes</b>						
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP Residential GCTL	23	1	0.3
TETRACHLOROETHENE	127-18-4	3	FDEP Residential GCTL	1.0	1	0.3
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FDEP Residential GCTL	33	1	0.3
TRICHLOROETHENE	79-01-6	3	FDEP Residential GCTL	1.0	1	0.3
VINYL CHLORIDE	75-01-4	1	FDEP Residential GCTL	0.33	1	0.3

- (1) The Project Action Limit is the Florida Administrative Code Chapter 62-777 Groundwater Cleanup Target Level (GCTL) for groundwater samples. April 2005.
- (2) The PALs presented for this compound is recognized by FDEP as not consistently achievable as per "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits". (FDEP, October 2004). For these compounds, the laboratory LOQ will serve as the PAL which is suggested by this guidance document.

**Abbreviations**

CAS = Chemical Abstracts Service

FDEP = Florida Department of Environmental Protection

GCTL= Groundwater Cleanup Target Level (Residential) ([http://www.dep.state.fl.us/waste/quick\\_topics/rules/documents/62-777/TableGroundwaterCTLs4-17-05.pdf](http://www.dep.state.fl.us/waste/quick_topics/rules/documents/62-777/TableGroundwaterCTLs4-17-05.pdf))

µg/L = micrograms per liter

NA = limit or goal not available for this analyte

**Notes:**

**Bolded rows indicate that the PAL is between the laboratory LOQ and MDL.**

**Bolded Shaded rows indicate that the PAL is at or below the laboratory MDL.**

**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 SAP Work Plan & Appendices	Tetra Tech	08/26/09	4/30/10		
<b>Submit Rough Draft SAP Work Plan &amp; Appendices</b>	Tetra Tech		4/30/10		
Navy Review	Navy	4/30/10	05/31/10		
Prepare Final SAP Work Plan & Appendices	Tetra Tech	05/31/10	06/15/10		
<b>Submit Final SAP Work Plan &amp; Appendices</b>	Tetra Tech		06/15/10		
Regulator Review	USEPA & FDEP	06/21/10	07/21/10		
Receive Comments/Comment Resolution	Tetra Tech	07/21/10	07/30/10		
<b>Mobilization and Field Investigation Event 1 Monitoring Well Installation</b>	Tetra Tech	08/02/10	08/11/10		
<b>Event 2- Groundwater Sampling</b>	Tetra Tech	08/16/10	08/25/10		
<b>Event 3- Surveying</b>	Tetra Tech	08/30/10	09/02/10		
<b>Event 4- IDW Management</b>	Tetra Tech	09/02/10	09/06/10		
<b>Complete Field Investigation and Demobilization</b>	Tetra Tech		09/06/10		
<b>Laboratory Analysis</b>	Empirical	08/16/10	09/30/10		
<b>Data Validation</b>	Tetra Tech	08/31/10	10/10/10		
Database Entry	Tetra Tech	08/31/10	10/10/10		
Prepare Rough Draft RI	Tetra Tech	10/10/10	12/10/10		
<b>Submit Rough Draft RI</b>	Tetra Tech		12/10/10		
Navy Review Draft RI	Navy	12/10/10	01/10/11		
Prepare Draft Final RI	Tetra Tech	01/10/11	01/17/11		
<b>Submit Draft Final RI</b>	Tetra Tech		01/17/11		

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Regulator Review	USEPA & FDEP	01/17/11	02/17/11		
Receive Comments/Comment Resolution	Tetra Tech	02/17/11	02/25/11		
Prepare Final RI	Tetra Tech	02/25/11	03/04/11		
<b>Submit Final RI</b>	Tetra Tech		03/04/11		

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

Groundwater data was generated in 2007 and 2008 by the Project Team to develop a series of data sets to help determine if the North Field plume was shrinking, stable, or expanding. After it was noted the North Field plume boundary was not complete, existing data was used to assist with the selection of additional monitoring well placement, which will be used to complete the boundary.

The selected judgmental sampling design is based on collecting representative samples from the environmental media of interest in areas that have a significant amount of data gaps from previous groundwater investigations conducted at Site 40. Sampling locations, identifications, and methods are listed in Worksheet #18. The target analytes including, BTEX, TCE+, and metals are included in each Analytical Group listed in Worksheet #15.

The investigation involves the collection of groundwater samples from biased locations identified on Figure 17-1. The following paragraphs describe the sampling approach.

A judgmental sampling design was selected to determine whether hazardous substances are present in the northern, eastern, and southern margin of the North Field plume at concentrations that exceed risk-based criteria. Seven locations were selected for installation of new monitoring wells and three existing monitoring well locations were selected for additional groundwater sampling and biased to the margin of the North Field plume to better define that boundary. A shallow surficial groundwater sample will be collected from approximately 110 feet bgs interval at each monitoring well location. Groundwater analytical data from the seven new monitoring wells will be added to the existing data set to determine the nature and extent of target analytes including BTEX, TCE, and metals concentrations at the North Field plume. Groundwater analytical data from the three existing monitoring wells will be added to the existing data set to determine the nature and extent of target analytes including BTEX and TCE+ (these wells have sufficient historical metals data so new data are not necessary at this time) concentrations at the North Field plume.

Groundwater analytical data from seven existing monitoring wells will be added to the existing data set to determine the nature and extent of BTEX concentrations at UST Site 1438/1439. Groundwater analytical data from two new and three existing monitoring wells will be added to the existing data set to determine the nature and extent of target analyte concentrations at Site 2948.

The NAS Whiting Field Partnering Team agreed that 10 samples, taken at biased locations where hazardous substances would likely be present in or on the edge of the plume, would be sufficient to make decisions on the path forward for the North Field plume area. Seven additional samples will be sufficient

to assess the current level of BTEX concentrations at UST Site 1438/1439. Five additional samples will be sufficient to assess the current level of target analytes concentrations at Site 2948.

In order to complete the field event for this investigation, nine groundwater monitoring wells will be installed utilizing Roto-Sonic technology. Roto-Sonic borings will be screened during groundwater monitoring well installation. Once the borings are complete, monitoring wells will be installed. The wells will be screened 20 feet across the water table. The new monitoring well locations at Site 40 were chosen by analyzing data from past investigations and determining where data gaps exist. Due to the existence of perched wells and wells that have been destroyed, new wells need to be installed in these locations to complete the remedial investigation.

Following the installation and development of the new wells, groundwater samples will be collected and analyzed from the 9 new monitoring wells and from 13 existing wells in accordance with FDEP SOP FS 2200. Sample locations were chosen to fill data gaps at the eastern boundary of the North Field plume, at UST Site 1438/1439 and at Site 2948. All samples will include the collection and measurements of the field parameters of DO, conductivity, pH, turbidity, and ORP, as well as elevation.



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

Sampling Location / ID Number <sup>1</sup>	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
New Wells – Eastern Boundary					
WHF-1467-MW-38S/ 1467G3801	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-39S/ 1467G3901	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-40S/ 1467G4001	GW	110	BTEX, TCE+, Metals	1 + field duplicate	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-41S/ 1467G4101	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-42S/ 1467G4201	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-43S/ 1467G4301	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1467-MW-44S/ 1467G4401	GW	110	BTEX, TCE+, Metals	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
Existing Wells – Eastern Boundary					
WHF-1467-MW-14S/ 1467G1401	GW	110	BTEX, TCE+	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-03-MW-4S/ 03G0401	GW	115	BTEX, TCE+	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-33-MW-5S/ 33G0501	GW	140	BTEX, TCE+	1	FS1000, FS2200 Tetra Tech SOP SA 1.1

<b>Sampling Location / ID Number<sup>1</sup></b>	<b>Matrix</b>	<b>Depth (feet bgs)</b>	<b>Analytical Group</b>	<b>Number of Samples (identify field duplicates)</b>	<b>Sampling SOP Reference</b>
<b>Existing Wells – Site 1438/1439</b>					
WHF-1438-MW-2D/ 1438G0203	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1438-MW-2S/ 1438G0201	GW	115	BTEX	1 + field duplicate	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1438-MW-3S/ 1438G0301	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1438-MW-5S/ 1438G0501	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1438-MW-6S/ 1438G0601	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-1438-MW-7S/ 1438G0701	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-05-MW-8S/ 05G0801	GW	115	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
<b>New Wells – Site 2894</b>					
WHF-2894-MW-3I/ 2894G0302	GW	120	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-2894-MW-4I/ 2894G0402	GW	120	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1

<b>Sampling Location / ID Number<sup>1</sup></b>	<b>Matrix</b>	<b>Depth (feet bgs)</b>	<b>Analytical Group</b>	<b>Number of Samples (identify field duplicates)</b>	<b>Sampling SOP Reference</b>
Existing Wells – Site 2894					
WHF-2894-MW-1/ 2894G0102	GW	114	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-2894-MW-2/ 2894G0202	GW	117	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1
WHF-2894-MW-7P/ 2894G0701	GW	87	BTEX	1	FS1000, FS2200 Tetra Tech SOP SA 1.1

<sup>1</sup>The ID number consists of, in order, the site number, G for groundwater, the monitoring well identification number, and 01 for shallow wells, 02 for intermediate wells, and 03 for deep wells. For example, WHF-33-MW-5S is 33G0501.

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

<b>Matrix</b>	<b>Analytical Group</b>	<b>Analytical and Preparation Method / SOP Reference</b>	<b>Containers (number, size, and type)</b>	<b>Sample Volume (units)</b>	<b>Preservation Requirements (chemical, temperature, light protected)</b>	<b>Maximum Holding Time (preparation/analysis)</b>
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 ( $\pm$ 2) °C; no headspace	14 days to analysis
Groundwater and aqueous QC blanks	Metals, Including Mercury	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 ( $\pm$ 2) °C	180 days to analysis except mercury, 28 days for mercury

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

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs (BTEX)	12	2	1/1	0	1	1	0	16
Groundwater	VOCs (BTEX and TCE+)	10	1	1/1	1	1	1	0	14
Groundwater	Metals	7	1	1/1	1	1	NA	0	10

MS/MSD = matrix spike/matrix spike duplicate

<sup>1</sup> Although the MS/MSD is not typically considered a field QC, and is not included in the Total No. of Samples to Lab, it is included here because location determination is often established in the field.

**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	TtNUS	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 29, 2001	TtNUS	NA	N	Contained in Appendix A.
DV-01	Data Validation-Non-CLP Organics for Solid and Aqueous Matrices, Revision 1, 2/2/2009	TtNUS	NA	N	Contained in Appendix A.
DV-04	Data Validation-Non-CLP Inorganics for Solid and Aqueous Matrices, Revision 1, 8/13/2001	TtNUS	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.
FS 1000	General Sampling Procedures, 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	Included in FDEP SOP FS 2200. 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.

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 1200	Field Specific Conductance, December 2008	FDEP	Multi-parameter water quality meter, such as a Horiba U-22	N	Contained in Appendix A.
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	TtNUS	NA	N	Contained in Appendix A.
GH-1.3	Soil and Rock Drilling Methods, Revision 1, June 1999	TtNUS	NA	N	Contained in Appendix A.
GH-1.5	Borehole and Sample Logging, Revision 1, June 1999	TtNUS	NA	N	Contained in Appendix A.
HS-1.0	Utility Locating and Excavating Clearance, Revision 2, December 2003	TtNUS	Soil clearance equipment	N	Contained in Appendix A.
PCS-006	Design, Installation, and Placement of Monitoring Wells	FDEP	Hollow Stem Augers, Drill rods, flush joint casing	N	Contained in Appendix A.
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing, Revision 7, 4/7/2008	TtNUS	Pump equipment, water quality meter	N	Contained in Appendix A.
SA-6.1	Non-Radiological Sample Handling Revision 3, February 2004	TtNUS	Sample Bottle Ware, Packaging Material, Shipping Materials	N	Contained in Appendix A.
SA-6.3	Field Documentation Revision 3, March 2009	TtNUS	Field Logbook, Field Sample Forms, Boring Logs	N	Contained in Appendix A.

<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	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
Water Quality Meter	Visual Inspection  Calibration/ Verification	Daily  Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	FOL or designee	Manufacturer's Guidance Manual	None
Turbidity Meter	Visual Inspection  Calibration/ Verification	Daily  Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	FOL or designee	Manufacturer's Guidance Manual	None
Electric Water Level Indicator	Visual Inspection  Field checks as per manufacturer	Daily  Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	FOL or designee	Manufacturer's Guidance Manual	None
Flame Ionization Detector	Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL or designee	Manufacturer's Guidance Manual	None

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

<b>Lab SOP Number</b>	<b>Title, Revision Date, and / or Number</b>	<b>Definitive or Screening Data</b>	<b>Matrix and Analytical Group</b>	<b>Instrument</b>	<b>Organization Performing Analysis</b>	<b>Modified for Project Work? (Y/N)</b>
Empirical SOP-100	Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C (Revision 19, 4/20/09)	Definitive	Groundwater and aqueous QC blanks/ Metals digestion	NA/ Preparation	Empirical	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique Methods SW846 7470A & 245.1, CLP-M 4.1 (Revision 16, 1/28/09)	Definitive	Groundwater, surface water, and aqueous QC blanks/ Mercury	Flow Injection Mercury Analyzer	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 15, 5/08/09)	Definitive	Groundwater and aqueous QC blanks/ Metals	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Empirical	N
Empirical SOP-202	GC/MS Volatiles by Method 624 and SW846 Method 8260B (Revision 22, 9/30/09)	Definitive	Groundwater and aqueous QC blanks/ VOCs	GC/MS	Empirical	N

Copies of all Laboratory SOPs are provided in Appendix C.

**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) – Minimum of a 5-point calibration curve is prepared for all target analytes.	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification.	The average response factor (RF) for System Performance Check Compound (SPCCs) must be $\geq 0.30$ for chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq 0.1$ for chloromethane, bromoform, and 1,1-dichloroethane. The relative standard deviation (RSD) for RFs for calibration check compounds (CCCs) for must be $\leq 30\%$ ; and RSD for each analyte must be $\leq 15\%$ , or the linear least squares regression (r) must be $\geq 0.995$ ; or the coefficient of determination ( $r^2$ ) must be $\geq 0.99$ .	Repeat calibration if criterion is not met.	Analyst, Department Manager	Empirical SOP-202
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) of all analytes must be within 80-120% of true value.	Correct problem and verify second source standard. Reanalyze ICAL.	Analyst, Department Manager	
	Retention Time Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within $\pm 0.006$ RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	Continuing Calibration Verification (CCV)	Perform one per 12-hour analysis period after tune.	The RF for SPCCs must be $\geq 0.30$ for chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq 0.1$ for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target compounds and surrogates must be $\leq 20\%$ .	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 - Bromofluorobenzene (BFB)	At the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Retune and/or clean source.	Analyst, Department Manager	
ICP Metals	ICAL - the instrument is calibrated by a 1-point calibration per manufacturer's guidelines.	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	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 > 2x MDL.	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	

<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>
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > LOQ.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst, Department Manager	
	Low-Level Check Standard (if using 1-point ICAL)	Daily after 1-point ICAL and before samples.	The %R 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 of an analytical run.	ICS A recoveries must be within the absolute value of the LOQ; and ICS B recoveries must be within 80-120 %R of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
Mercury	ICAL – A 5-point calibration curve is prepared.	Perform daily prior to sample analysis.	The RSD for RFs must be ≤20%, or r must be ≥ 0.995.	Recalibrate.	Analyst, Department Manager	Empirical SOP-103
	ICV – Second Source	Each analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst, Department Manager	
	Calibration Blank	One is performed at the beginning of analytical sequence, after every 10 samples, at the end of the sequence.	The target analyte concentration must be < LOQ.	Re-prepare and analyze all associated samples.	Analyst, Department Manager	
	CCV	Perform every 10 samples and at the end of the analytical sequence.	The %R must be within 80-120% of the true value.	Recalibrate.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	CCV (undistilled)	CCV (undistilled)-at beginning and end of each run sequence and every 10 samples.	The %R must be within 90-100% of the true value.	If the CCV (undistilled) fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

**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	Acceptanc e Criteria	Corrective Action	Responsible Person	SOP Reference <sup>(1)</sup>
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in 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
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.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP-100/105
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab 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

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

<b>SAMPLE COLLECTION, PACKAGING, AND SHIPMENT</b>
Sample Collection (Personnel/Organization): FOL/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL/ Tetra Tech
Type of Shipment/Carrier: Federal Express
<b>SAMPLE RECEIPT AND ANALYSIS</b>
Sample Receipt (Personnel/Organization): Sample Custodians/Empirical
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab / Empirical
Sample Determinative Analysis (Personnel/Organization): GC/MS Lab, Metals Lab/ Empirical
<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): N/A
<b>SAMPLE DISPOSAL</b>
Personnel/Organization: Waste Disposal Technician/ Empirical
Number of Days from Analysis: 30 days from submittal of final report or 60 days from receipt, whichever is longer

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

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.

### **27.1 SAMPLE NOMENCLATURE**

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

### **27.2 SAMPLE COLLECTION 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 utilized for this project. All pages of each logbook will be numbered sequentially, and observations will be recorded with indelible ink.

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

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 of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities associated with sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, and arrival and departure times
- Health and safety issues including PPE

### **27.3 SAMPLE PACKAGING AND SHIPPING**

Sample packaging and shipping procedures presented in FDEP SOP FS1000 (Appendix A) will be followed. Sample log sheets will be prepared for each sample collected and will include sample-specific information, as well as information that documents sampling activities. Sample log sheets will be signed and dated, and the appropriate chain-of-custody procedures will be followed until the samples reach the analytical laboratory.

Samples will be prepared for shipping using the following guidelines:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g., Ziplock baggie), and seal bag.
- Place sample in cooler constructed of sturdy material which has been lined with a large plastic bag (e.g., “garbage bag”). Drain plugs on coolers should be taped shut.
- A temperature check indicator provided by the laboratory should be placed 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, place ice around sample container shoulders, and on top of packing material to adequately cool sample to 4°C.
- Seal (i.e., tape or tie top in knot) large liner bag.
- The original top, signed copy of the chain of custody form shall be placed in a large Ziploc-type bag and taped 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 state how many coolers are included with the shipment.
- Close and seal 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 marking is required.

## **27.4 SAMPLE HANDLING AND TRACKING SYSTEM**

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

## **27.5 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.

A sample is under custody if:

- It is in your actual possession, or
- It is in your view, after being in your physical possession, or
- It was in your possession and then you locked or sealed it up to prevent tampering, or
- It is in a secure area.

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

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

### **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 (contained in 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 in order 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 collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This form documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs which are included in Appendix C.

### **Laboratory Custody**

Custody Seals are supplied with all bottle orders. They are affixed to the cooler after sampling. The presence or absence of Custody Seals is noted on the Sample Receipt Condition Report (SRCR).

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

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

A laboratory data file is also initiated for the work order. This file includes the Login Chain of Custody, chain-of-custody and SRCR. The folder also includes a Login File Sheet which summarizes the analyses that the work order has been logged for. This sheet is used to track data completion.

Samples for a project may be batched or grouped together by the laboratory. A series of batched work orders is referred to as a SDG. The SDG includes those samples received on a chain of custody, duplicate samples, and field QA/QC samples, and can include samples of different media. QA/QC

samples will be run at the frequency specified in the analytical methods. The SDG is given a specific identification number.

Samples are stored at the laboratory in refrigerators prior to, during, and after analysis. Refrigerators at the laboratory are constantly monitored for temperature. Proper temperatures and lighting are maintained in the refrigerators to ensure sample integrity and preservation. Samples are retained by the laboratory for a period of 90 days after the data report is mailed to the client unless otherwise specified in a client contract. The laboratory then disposes of non-hazardous samples, following certified disposal practices. Hazardous samples are either returned to the client or disposed of through a licensed broker. Documentation of disposal is maintained by the laboratory.

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

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

Matrix	Groundwater and Aqueous QC Blanks					
Analytical Group	VOCs					
Analytical Method/ SOP Reference	SW-846 8260B Empirical SOP-202					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One 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	Empirical %R limits (Appendix C). %R: Water Dibromofluoromethane 85-120 1,2-dichloroethane-d4 80-135 Toluene-d8 85-115 BFB 85-120	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	Groundwater and Aqueous QC Blanks					
Analytical Group	VOCs					
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP-202					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One per SDG or every 20 samples.	Empirical %R limits (Appendix C). RPD should be $\leq 30\%$ .	Corrective actions will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias / Precision	Same as Method/SOP QC Acceptance Limits..
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) (not required)	One is performed for each batch of up to 20 samples.	Empirical %R limits (Appendix C). RPD must be $\leq 30\%$ (for LCS/LCSD).	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	Retention times for ISs must be within $\pm 30$ seconds and the response areas must be within -50% to +100% of last CCV 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.

Matrix	Groundwater and Aqueous QC Blanks					
Analytical Group	Metals, Including Mercury					
Analytical Method / SOP Reference	SW-846 6010B Empirical SOP-100/103/105					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples.	No analytes detected > ½ the LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value; then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	Empirical %R limits (Appendix C). RPD ≤30% (for LCS/LCSD). Water: 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 Precision also, if LCSD is analyzed	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 ≤20%.	Narrate any results that are outside control limits.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
MS	One per 20 samples of similar matrix.	Empirical %R limits (Appendix C). The %R should be within 80-120%, if sample < 4x spike added.	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.

Matrix	Groundwater and Aqueous QC Blanks					
Analytical Group	Metals, Including Mercury					
Analytical Method / SOP Reference	SW-846 6010B Empirical SOP-100/103/105					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution	One is performed for each preparation batch with sample concentration(s) > 50x MDL.	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 MDL.	The result must agree within $\pm 25\%$ of expected result.	Flag results of samples of same matrix as estimates in SDG narrative.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

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

Document	Location Maintained
<p>Sample Collection Documents and Records:</p> <ul style="list-style-type: none"> <li>• Field logbook (and sampling notes)</li> <li>• Field sample forms</li> <li>• Chain-of-custody records</li> <li>• Sample shipment airbills</li> <li>• Equipment calibration logs</li> <li>• Photographs</li> <li>• FTMR forms</li> <li>• SAP</li> <li>• Field Sampling SOPs</li> </ul> <p>Laboratory Documents and Records:</p> <ul style="list-style-type: none"> <li>• Sample receipt/login form</li> <li>• Sample storage records</li> <li>• Sample preparation logs</li> <li>• Sample analysis run logs</li> <li>• Corrective Action (CA) forms</li> <li>• Reported results for standards, QC checks, and QC samples</li> <li>• Data completeness checklists</li> <li>• Extraction/cleanup records</li> <li>• Raw data</li> </ul> <p>Data Assessment Documents and Records:</p> <ul style="list-style-type: none"> <li>• Field Sampling Audit Checklist (if an audit is conducted)</li> <li>• Analytical Audit Checklist (if an audit is conducted)</li> <li>• Data Validation Memoranda</li> <li>• Remedial Investigation Report</li> </ul> <p>Other Documents:</p> <ul style="list-style-type: none"> <li>• All versions of UFP-SAP</li> <li>• All letter and e-mail correspondence with regulatory agencies, including approvals and comments</li> <li>• Field Investigation data packages</li> </ul> <p>All versions of project reports</p>	<p>Tetra Tech project file; results will be provided in the Remedial Investigation Report.</p> <p>Tetra Tech project file; long-term data package storage at third party commercial document storage firm.</p> <p>Tetra Tech project file. All reports for Site 40 Basewide Groundwater will be stored in hardcopy in the Tetra Tech Tallahassee project file and electronically in the Tetra Tech server library.</p>

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

<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 and Aqueous QC Blanks	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Kim Kostzer <a href="mailto:kkostzer@empirlabs.com">kkostzer@empirlabs.com</a> Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	NA
	Metals (Including Mercury)		SW-846 6010B SW-846 7470A			

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

<b>Assessment Type</b>	<b>Frequency</b>	<b>Internal or External</b>	<b>Organization Performing Assessment</b>	<b>Person(s) Responsible for Performing Assessment</b> (title and organizational affiliation)	<b>Person(s) Responsible for Responding to Assessment Findings</b> (title and organizational affiliation)	<b>Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)</b> (title and organizational affiliation)	<b>Person(s) Responsible for Monitoring Effectiveness of CA</b> (title and organizational affiliation)
Field Systems Audit	1 per contract year	Internal	Tetra Tech	Person assigned by Tetra Tech QAM	PM and FOL, Tetra Tech	Auditor and PM, Tetra Tech	CLEAN QAM, Tetra Tech
Laboratory System Audit <sup>1</sup>	2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QA Manager or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical

1 Empirical has successfully completed the laboratory assessment process required as part of the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM, Version 4.1) under the DoD ELAP by a recognized Accrediting Body. The DoD ELAP accreditation letter is included in Appendix C.

**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 Corrective Action Response Documentation</b>	<b>Individual(s) Receiving Corrective Action Response</b> (name, title, organization)	<b>Timeframe for Response</b>
Field Sampling System Audit <sup>(1)</sup>	Audit checklist (as per Navy Installation Restoration Chemical Data Quality Manual [IRCDQM]) and written audit report	Rich May, PM, Tetra Tech; Larry Smith, FOL, Tetra Tech; Debra Humbert Program Manager, Tetra Tech; Chris Pike, Deputy Program Manager, Tetra Tech	Dependent on the finding; if major a stop work may be issued immediately; however, if minor within 1 week of audit	Written memo	Rich May, PM, Tetra Tech; Larry Smith, FOL, Tetra Tech; Debra Humbert Program Manager, Tetra Tech; Chris Pike, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Randy Ward, Laboratory QAM, Empirical	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

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

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

<b>Type of Report</b>	<b>Frequency</b> (daily, weekly monthly, quarterly, annually, etc.)	<b>Projected Delivery Date(s)</b>	<b>Person(s) Responsible for Report Preparation</b> (title and organizational affiliation)	<b>Report Recipient(s)</b> (title and organizational affiliation)
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data	DVM or designee, Tetra Tech	PM and project file, Tetra Tech
Major analysis problem identification (Internal, Tetra Tech Memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	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 the project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical	PM and project file, Tetra Tech

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

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	The Empirical Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Empirical 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and 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
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

<b>Verification Input</b>	<b>Description</b>	<b>Internal / External</b>	<b>Responsible for Verification</b> (name, organization)
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 MDL 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
	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; page 110 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

<b>Step IIa / IIb</b>	<b>Validation Input</b>	<b>Description</b>	<b>Responsible for Validation</b> (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	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.	Project Chemist or Data Validators, Tetra Tech
		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.	
		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.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	

<b>Step IIa / IIb</b>	<b>Validation Input</b>	<b>Description</b>	<b>Responsible for Validation</b> (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	<p>Sensitivity - Ensure that the project LOQs listed in Worksheet #15 were achieved.</p> <p>PALs - Discuss the impact on reported MDLs 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 PALs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PALs 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) (Figure 37; page 110 UFP-QAPP Manual)

<b>Step IIa / IIb</b>	<b>Matrix</b>	<b>Analytical Group</b>	<b>Validation Criteria</b>	<b>Data Validator</b> (title and organizational affiliation)
IIa and IIb	Groundwater and Aqueous QC Blanks	VOCs	SW-846 8260B method specific criteria, DoD QSM, and those criteria listed in Worksheets #12, #15, #24, and #28 will be used. If not included in Worksheet #12, #15, #24 or #28, 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	Groundwater and Aqueous QC Blanks	Metals (Including Mercury)	SW-846 6010B method specific criteria, DoD QSM, and those listed in Worksheets #12, #15, #24, and #28 will be used. If not included in Worksheet #12, #15, #24, and #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

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

**Data Usability Assessment**

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

**Completeness**

- For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the project team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If 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, MS, and LCSs. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

**Representativeness**

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

**Comparability**

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

**Sensitivity**

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

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

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

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

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

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, 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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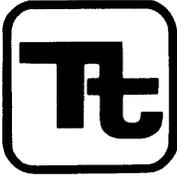
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**APPENDIX A**

**TETRA TECHNUS, INC. AND  
FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION  
STANDARD OPERATING PROCEDURES**



TETRA TECH NUS,  
INC.

# STANDARD OPERATING PROCEDURES

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Effective Date	03/09/09	Revision	2
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston		

Subject  
SAMPLE NOMENCLATURE

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## 1.0 PURPOSE

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

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

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

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

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

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

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

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## 5.0 PROCEDURES

### 5.1 INTRODUCTION

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

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

Additional segments may be added as needed. For example:

- (1) Soil and sediment sample ID

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

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

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

- (3) Biota sample ID

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

### 5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

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

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

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

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

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

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

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

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

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

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

### 5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

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

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

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

Sample type - Examples of sample types are as follows:

- AH - Ash Sample

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

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

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

Species identifier - Examples of species identifier are as follows:

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

#### 5.4 EXAMPLES OF SAMPLE NOMENCLATURE

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

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

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

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

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

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

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

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

## 5.5 FIELD QA/QC SAMPLE NOMENCLATURE

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

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

The QC types are identified as:

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

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

## 5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

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

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

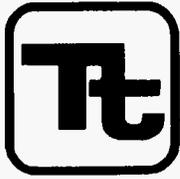
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The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

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

## **6.0 DEVIATIONS**

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



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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

Subject  
DATABASE RECORDS AND QUALITY ASSURANCE

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## 1.0 PURPOSE

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

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

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

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

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

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

## 4.0 RESPONSIBILITIES

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

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

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

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

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

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

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

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

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

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

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

## 5.0 PROCEDURES

### 5.1 Introduction

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

### 5.2 File Establishment

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

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

### 5.3 Electronic Deliverables

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

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#### 5.4 Sample Tracking Forms

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

#### 5.5 Chain-of-Custody Forms

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

#### 5.6 Data Validation Letters

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

#### 5.7 Historical Data

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

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

## 6.0 RECORDS

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

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

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

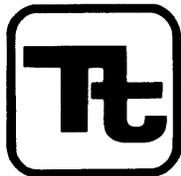
**ATTACHMENT A**



**MIS REQUEST FORM**

Tetra Tech NUS, Inc.

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



TETRA TECH NUS,  
INC.

# STANDARD OPERATING PROCEDURES

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Effective Date 02-02-09	Revision 1
Applicability Tetra Tech NUS, Inc.	
Prepared Chemistry and Toxicology Department	

Subject  
DATA VALIDATION – NON-CLP ORGANICS FOR  
SOLID AND AQUEOUS MATRICES

Approved  
T. Johnston *T.E. Johnston*

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**APPENDIX A - SAMPLE CALCULATIONS (excerpted from EPA SOM01.1)**

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## 1.0 PURPOSE

This SOPC governs the validation of data generated by the following methods:

- Gas Chromatography/Mass Spectrometry
  - Volatile Organic Compounds (VOCs) by METHOD 8260B
  - Semivolatile Organic Compounds (SVOCs) by METHOD 8270C
- Gas Chromatography
  - Volatile Organic Compounds (VOCs) by SW 5030/SW 8011/8015B/8021A/8031
  - Semivolatile Organic Compounds (SVOCs) by SW 8041/8061A/8091/8310
  - Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides by SW 8081A/8082/8141A/8151A

## 2.0 APPLICABILITY

The applicability of each set of validation criteria is described in the appropriate section below.

## 3.0 PERSONNEL QUALIFICATIONS

The minimum qualifications of persons implementing this SOP are as follow:

- Education – Minimum of a bachelor’s degree in chemistry or related physical/life science.
- Experience requirements include either operational experience with the analytical method or method data review training conducted under the direction of an experienced reviewer and performed on the subject matter data package. A record of the training will not be documented and kept on file but the data validation report produced under training will serve as the record.

## 4.0 CLP ORGANICS BY GC/MS

### 4.1 Volatiles (METHOD 8260B)

#### 4.1.1 Applicability

Method 8260B is used to determine volatile organic compounds in most waste matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 8260B analyte list includes of the volatile Target Compound List (TCL) plus the following compounds\*:

Acetonitrile	trans-1,2-Dichloroethene
Acrolein	Ethyl methacrylate
Acrylonitrile	Iodomethane
Allyl chloride	Methacrylonitrile
Chloropropene	Methyl methacrylate
1,2-Dibromo-3-chloropropane	2-Picoline
1,2-Dibromoethane	Pyridine
Dibromomethane	Trichlorofluoromethane
trans-1,4-Dichloro-2-butene	1,2,3-Trichloropropane
Dichlorodifluoromethane	Vinyl acetate

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\* Appendix IX target compounds

Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared by Method 5030.

#### 4.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

#### 4.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

#### 4.1.4 Sample Preparation

Method 5030 is a purge-and-trap procedure performed to prepare and extract volatile compounds from samples and introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

#### 4.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

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Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

#### **4.1.6 Technical Evaluation Summary**

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

##### **4.1.6.1 Holding Times and Sample Preservation Criteria**

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

- a. The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.
- b. No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.
- c. For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

##### **4.1.6.2 Holding Time and Sample Preservation Action**

- a. Positive results in affected samples are generally qualified as estimated (J); non-detects (UJ). These results are biased low.
- b. Some USEPA Regions apply the bias qualifiers, L and UL, instead.
- c. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); non-detects are generally considered to be unreliable and are qualified (R).
- d. Results for which the holding time was grossly exceeded are biased low.

##### **4.1.6.3 GC/MS Tuning Criteria**

An analysis of an instrument performance check standard of Bromofluorobenzene must be performed at the beginning of each 12-hour period in which samples and standards are being analyzed.

- a. Verify that all ion abundance criteria below are within acceptance ranges on Form V or equivalent summary form:

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m/z	Ion abundance criteria
50	8.0 – 40.0% of m/z 95
75	30.0 – 66.0% of m/z 95
95	Base peak, 100%
96	5.0 – 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 – 120.0% of m/z 95
175	5.0 – 9.0% of m/z 174
176	93.0 – 101.0% of m/z 174
177	5.0 – 9.0% of m/z 176

- b. Verify that all samples and standards were analyzed within the 12-hour period.

#### 4.1.6.4 GC/MS Tuning Action

- a. If mass assignment is in error, then reject all associated data (R) or (UR).
- b. If ion abundance criteria are not met, professional judgment may be used to determine the extent of data usability and whether qualifications are needed. The most critical abundances are m/z 95/96, 174/175, 174/176, and 176/177.
- c. If samples were analyzed beyond the 12-hour period, then qualify positive and non-detected results as estimated, (J) and (UJ) respectively.
- d. If the reviewer suspects that improper background subtraction techniques were used to generate a compliant tune, contact the laboratory and ask them to provide supporting evidence of tuning data. If the evidence is suitable, no further action is required. If proper evidence cannot be provided to support the tuning data, then professional judgment should be utilized to determine the usability of the associated data.

#### 4.1.6.5 Calibration Criteria

Verify the following:

- a. Verify that an initial calibration was performed for each instrument used for analysis and for each type of medium and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.
- b. Review the data package Form Vs (tuning) using the applicable USPEA Regional Functional Guidelines, and qualify the data as appropriate.
- c. Review initial calibration Form VIs and the associated laboratory raw data to determine which compounds have:
1. average Relative Response Factors (RRFs) <0.050
  2. Percent Relative Standard Deviations (%RSDs) >30%.
- d. Circle noncompliant RRFs and %RSDs on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.
- e. Determine which samples are affected by non-compliant RRFs or %RSDs by reviewing the continuing calibration Form VIIs. Check the instrument identification and the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations.

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- f. Review the sample listings given on the data package Form Vs to match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers.
- g. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII.
- h. Review the continuing calibration Form VIIs and the associated laboratory raw data to determine which compounds have:
  1. RRFs <0.050
  2. Percent Differences (%Ds) >25%
- i. Circle the noncompliant RRFs and %Ds on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

#### **4.1.6.6 Calibration Actions**

- a. If any RRFs are <0.050, qualify all affected positive as estimated (J); qualify non-detects as non-detected rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- b. If any %RSD exceeds 30%, qualify affected positive results as estimated (J); qualify non-detects as non-detected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate non-detects if the %RSD is >50% or reject non-detects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- c. If any %D exceeds 25%, qualify affected positive results as estimated (J); qualify non-detects as non-detected estimated (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate non-detects if the %D is >50% or reject non-detects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.

#### **4.1.6.7 Blank Contamination Criteria**

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

#### **4.1.6.8 Blank Contamination Action**

- a. If a target compound is detected in any method blank:
  1. Select the maximum concentration of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!)
  2. Establish action levels for qualification (10X or 5X the maximum contaminant concentration depending upon whether or not the contaminant is a common contaminant). Common laboratory contaminants may vary among protocols.

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3. Raise positive results that are less than the established blank action level to the Contract Required Quantitation Limit (CRQL) and qualify them as non-detect (U). In accordance with some USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results. In this case, qualify the results at the concentration detected instead of the CRQL.

- b. If a target compound was detected in a field quality control blank, carefully evaluate the associated samples to determine the appropriate action. Typically, field quality control blanks are not used to establish blank action levels but professional judgment may be used. When the reviewer decides to use a field quality control blank to qualify associated environmental samples, the guideline above must be followed.

#### **4.1.6.9 Surrogates Criteria**

- a. Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.
- b. Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any non-compliance on your working copies of these Forms.

#### **4.1.6.10 Surrogate Action**

- a. Results for all compound in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided.
- b. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), non-detects are rejected (UR). These results are biased low.
- c. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); non-detects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- d. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance, non-detects are not qualified based on high surrogate recovery.

#### **4.1.6.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria**

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

#### **4.1.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action**

- a. No action is generally taken on MS/MSD non-compliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results as estimated (J) and qualify non-detects as non-detected rejected (UR) in the unspiked sample.

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#### **4.1.6.13 Internal Standard Criteria**

Internal standards are evaluated by reviewing the data package Form VIIIs and laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any non-compliance on your working copies of these forms.

#### **4.1.6.14 Internal Standard Action**

Evaluate and qualify as stipulated in the appropriate data validation protocol.

#### **4.1.6.15 Tentatively Identified Compounds (TICs) Criteria**

Verify that the laboratory reported TICs in the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

#### **4.1.6.16 Field Duplicate Precision Criteria**

- a. Check samples to determine if field duplicates were included in the data package.
- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50% for sample results greater than the reporting limit.

#### **4.1.6.17 Field Duplicate Precision Action**

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Qualify positive results showing imprecision as estimated (J). Bias for these results cannot be determined.
- b. If one result is positive and the other is non-detected and the positive result is greater than 2 times the reporting limit, qualify positive and non-detected results as estimated (J) or non-detected estimated (UJ), respectively.

#### **4.1.6.18 Sample Result Verification Criteria**

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

#### **4.1.6.19 Sample Result Verification Action**

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

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#### **4.1.6.20 Percent Solids Criteria**

Check the percent solids for each sample to identify any samples that contain <30% solids.

#### **4.1.6.21 Percent Solids Action**

- a. If any sample contains <30% solids, qualify positive and non-detected results as estimated (J) or non-detected estimated (UJ), respectively, due to the high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); qualify non-detected results as rejected (UR).

#### **4.1.6.22 Laboratory Precision Criteria**

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds.

#### **4.1.6.23 Laboratory Precision Action**

Consider non-detects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

#### **4.1.7 Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

### **4.2 Semivolatiles (METHOD 8270C)**

#### **4.2.1 Applicability**

Methods are applicable to most types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

These methods can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of elution without derivatization as sharp peaks from a gas chromatographic column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The above methods specifically analyze for the semivolatile Target Compound List (TCL) plus the following compounds\*:

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Acetophenone	Hexachlorophene	N-nitrosodimethylethylamine
Aniline	Hexachloropropene	N-nitroso-di-n-butylamine
Benzyl alcohol	Isodrin	N-nitrosomorpholine
Bis(2-chloroisopropyl)ether	Isosafrole	N-nitrosopiperidine
Chlorobenzilate	Kepone	Pentachlorobenzene
Diallate	Methapyrilene	Pentachloronitrobenzene
2,6-Dichlorophenol	3-Methylcholanthrene	Phenacetin
Dimethoate	Methyl methanesulfonate	p-Phenylenediamine
p-Dimethylaminoazobenzene	3-Methylphenol	Phorate
7,12-Dimethylbenz(a)anthracene	1,4-Naphthoquinone	2-Picoline
3,3'-Dimethylbenzidine	4-Nitroquinoline-1-oxide	Pronamide
a,a-Dimethylphenylamine	1-Naphthylamine	Safrole
1,3-Dinitrobenzene	2-Naphthylamine	1,2,4,5-Tetrachlorobenzene
Diphenylamine	5-Nitro-o-toluidine	Thionazin
Ethyl methanesulfonate	N-nitrosodiethylamine	o,o,o-Triethylphosphorothioate
Famphur	N-nitrosodimethylamine	1,3,5-Trinitrobenzene

\* Appendix IX target compounds

The preceding method is based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures, Method 8270C uses GC/MS capillary column technique.

#### 4.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts that cause elevated baselines and lead to potential misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source depending upon the diversity of the industrial complex or waste being sampled.

#### 4.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted once per 20 samples of a similar matrix to determine the effects of sample matrix upon the compounds of interest.

#### 4.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or sonication (Method 3550) procedures.

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#### 4.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate numbers of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.
- c. Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better data quality.
- d. Prepare working copies of all Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

#### 4.2.6 Technical Evaluation Summary

Conduct all data evaluations in accordance with the appropriate USEPA Regional protocols (when applicable) and/or specified client contract requirements. Reference the applicable documents during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

Evaluate general parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification concurrently with the parameters discussed in the following subsections.

##### 4.2.6.1 Holding Times and Sample Preservation Criteria

Verify that holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Calculate holding times for extraction from date of collection to date of extraction. Verify that samples are stored to method requirements. Use the following rules:

- a. For aqueous samples, use a 7-day maximum holding time until extraction.
- b. For soil samples, use a 14-day maximum holding time until extraction.
- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.

##### 4.2.6.2 Holding Times and Sample Preservation Action

- a. If holding times are exceeded, qualify positive results in affected samples as estimated (J), non-detects (UJ). These results are usually assumed to be biased low unless prolonged storage causes a concentration increase, e.g., for degradation products which are also target analytes.
- b. If holding times are exceeded by a factor of more than two times the required time, qualify positive results as estimated (J), non-detects are rejected (UR). These exceedances are considered to be gross holding time exceedances.

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- c. Alternatively, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance.
- d. Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.

#### 4.2.6.3 GC/MS Tuning Criteria

An analysis of an instrument performance check standard of Decafluorotriphenylphosphine (DFTPP) must be performed at the beginning of each 12-hour period in which samples and standards are being analyzed.

- a. Verify that all ion abundance criteria below are within acceptance ranges on Form V or equivalent summary form:

m/z	Ion abundance criteria
51	30.0 – 80.0% of m/z 198
68	Less than 2.0% of m/z 198
69	Mass 69 relative abundance
70	Less than 2.0% of m/z 69
127	25.0 – 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base Peak 100%
199	5.0 – 9.0% of m/z 198
275	10.0 – 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 – 110.0% of m/z 198
443	15.0 – 24.0% of m/z 442

- b. Verify that all samples and standards were analyzed within the 12-hour period.

#### 4.2.6.4 GC/MS Tuning Action

- a. If mass assignment is in error, then reject all associated data (R) or (UR).
- b. If ion abundance criteria are not met, professional judgment may be used to determine the extent of data usability and whether qualifications are needed. The most critical abundances are m/z 199/198 and 442/443.
- c. If the relative abundance of m/z 365 is low or is zero this is an indication of an unsuitable instrument zero. Detection limits may be affected and non-detected results should be qualified (UJ).
- d. If samples were analyzed beyond the 12-hour period, then qualify positive and non-detected results as estimated, (J) and (UJ) respectively.
- e. If the reviewer suspects that improper background subtraction techniques were used to generate a compliant tune, contact the laboratory and ask them to provide supporting evidence of tuning

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data. If the evidence is suitable, no further action is required. If proper evidence cannot be provided to support the tuning data, then professional judgment should be utilized to determine the usability of the associated data.

#### 4.2.6.5 Calibration Criteria

Verify the following:

- a. Verify that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.
- b. Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.
- c. Review initial calibration Form VIs and the associated laboratory raw data to determine which compounds have:
  1. Average Relative Response Factors (RRFs) <0.050
  2. Percent Relative Standard Deviations (%RSDs) >30%.
- d. Circle these noncompliant RRFs and %RSDs on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.
- e. Determine which samples are affected by non-compliant RRFs or %RSDs by reviewing the continuing calibration Form VIIs. Check the instrument identification and the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers.
- f. Review the continuing calibration Form VIIs and the associated laboratory raw data to determine which compounds have:
  1. RRFs <0.050
  2. Percent Differences (%Ds) >25%
- g. Circle the noncompliant RRFs and %Ds on your working copies of these Forms and spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

#### 4.2.6.6 Calibration Actions

- a. If any RRFs are <0.050, qualify all affected positive results as estimated (J), non-detects are rejected (UR). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is very low.
- b. If any %RSD exceeds 30%, qualify all affected positive results as estimated (J), non-detects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which only estimate non-detects if the %RSD is >50% or reject non-detects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.
- c. If any %D exceeds 25%, qualify all affected positive results as estimated (J), non-detects (UJ). Check the specific applicable data validation protocol for further guidance as there are some

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protocols which only estimate non-detects if the %D is >50% or reject non-detects if the %RSD is excessive (e.g. >90%). Bias for these results cannot be determined.

#### **4.2.6.7 Blank Contamination Criteria**

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

#### **4.2.6.8 Blank Contamination Action**

- a. If a target compound is detected in any method blank:
  1. Select the maximum concentration of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!)
  2. Establish action levels for qualification (10X or 5X the maximum contaminant concentration depending upon whether or not the contaminant is a common contaminant).
  3. Raise positive results that are less than the established blank action level to the Contract Required Quantitation Limit (CRQL) and qualify them as non-detect (U). In accordance with some USEPA Regional protocol, the (B) qualifier may be used instead of (U) when qualifying positive results. In this case, qualify results at the concentration detected instead of the CRQL.
- b. If a target compound was detected in a field quality control blank, carefully evaluate the associated samples to determine the appropriate action. Typically, field quality control blanks are not used to establish blank action levels but professional judgment may be used. When the reviewer decides to use a field quality control blank to qualify associated environmental samples, the guideline above must be followed.

#### **4.2.6.9 Surrogates Criteria**

Semivolatile compounds are divided into two fractions, base-neutral compounds and acid-extractable compounds. Each fraction of compounds has its own associated surrogates.

- a. Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.
- b. Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any non-compliance on your working copies of these Forms.

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#### 4.2.6.10 Surrogate Action

- a. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and non-detects are rejected (UR). There results are biased low.
- b. No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.
- c. If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); non-detects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead.
- d. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; non-detects are not qualified based on high surrogate recoveries.

#### 4.2.6.11 Matrix Spike/Matrix Spike Duplicate Criteria

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

#### 4.2.6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. Take no action based on MS/MSD non-compliances alone.
- b. If qualification does occur, generally only the results for that particular noncompliant compound are qualified in the original unspiked sample analysis. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

#### 4.2.6.13 Internal Standard Criteria

Evaluate internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. Circle any non-compliance on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

#### 4.2.6.14 Internal Standard Action

Evaluate and qualify as stipulated in the appropriate protocol.

#### 4.2.6.15 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I BNA-TIC reports and the laboratory raw data. The guidance given in the 3/90 national Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

#### 4.2.6.16 Field Duplicate Precision Criteria

- a. Check samples to determine if field duplicates were included in the data package.

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- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50% for sample results greater than the reporting limit.

#### 4.2.6.17 Field Duplicate Precision Action

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Qualify positive results for compounds showing imprecision are qualified as estimated (J). Bias for these results cannot be determined.
- b. If one result is positive and the other is non-detected and the positive result is greater than 2 times the reporting limit, qualify positive and non-detected results as estimated (J) and or non-detected estimated (UJ), respectively.

#### 4.2.6.18 Sample Result Verification Criteria

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

#### 4.2.6.19 Sample Result Verification Action

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

#### 4.2.6.20 Percent Solids Criteria

Check the percent solids for each sample to identify any samples that contain <30% solids.

#### 4.2.6.21 Percent Solids Action

- a. If any sample contains <30% solids, qualify positive and non-detected results as estimated (J) and non-detected (UJ), respectively, due to the high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); and non-detects are rejected (UR).

#### 4.2.6.22 Laboratory Precision Criteria

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results.

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#### 4.2.6.23 Laboratory Precision Action

Consider non-detects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

#### 4.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

### 5.0 SW-846NON-CLP ORGANICS BY GAS CHROMATOGRAPHY

#### 5.1 Volatiles (SW 5030/SW 8011/8051B/8021A/8031)

##### 5.1.1 Applicability

Method 8011 is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

1,2-Dibromoethane (EDB)  
1,2-Dibromo-3-chloropropane (DBCP)

Method 8021A is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Allyl chloride	4-Chlorotoluene	Methyl iodide
Benzyl chloride	Dibromochloromethane	1,1,2,2-Tetrachloroethane
Bis (2-chloroethoxy)methane	1,2-Dibromo-3-chloropropane	1,1,1,2-Tetrachloroethane
Bis (2-chloroisopropyl)ether	Dibromomethane	Tetrachloroethylene
Bromoacetone	1,2-Dichlorobenzene	1,1,1-Trichloroethane
Bromobenzene	1,3-Dichlorobenzene	1,1,2-Trichloroethane
Bromodichloromethane	1,4-Dichlorobenzene	Trichloroethylene
Bromoform	Dichlorodifluoromethane	Trichlorofluoromethane
Bromomethane	1,1-Dichloroethane	1,2,3-Trichloropropane
Carbon tetrachloride	1,2-Dichloroethane	Vinyl chloride
Chlorobenzene	1,1-Dichloroethylene (Vinylidene chloride)	Benzene
Chloroethane	trans-1,2-Dichloroethylene	Chlorobenzene
2-Chloroethanol	Dichloromethane	1,2-Dichlorobenzene
Chloroform	1,2-Dichloropropane	1,3-Dichlorobenzene
1-Chlorohexane	1,3-Dichloro-2-propanol	1,4-Dichlorobenzene
2-Chloroethyl vinyl ether	cis-1,3-Dichloropropene	Toluene
Chloromethane	trans-1,3-Dichloropropene	Ethyl benzene
Chloromethyl methyl ether	Epichlorhydrin	Xylenes (Dimethyl benzenes)
Chloroprene	Ethylene dibromide	

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Method 8015B is used to determine the concentration of the following nonhalogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Diethyl ether	Acrolein	n-butyl Alcohol
Ethanol	Acetonitrile	t-butyl Alcohol
Methyl ethyl ketone (MEK)	Acetone	Methanol
Methyl isobutyl ketone (MIBK)	Allyl Alcohol	1,4-Dioxane

Method 8031 is used to determine the concentration of the following volatile organic compound in groundwater, liquid, and solid matrices:

Acrylonitrile

All of the above Methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). Method 8021A analyzes for halogenated and aromatic volatile organics via GC/HECP and GC/PID (Electro Conductivity Detector and Photoionization detector), Method 8015B analyzes for non-halogenated volatile organics via GC/FID (Flame Ionization Detector), and Method 8031 analyzes for the compounds acrylonitrile using GC/FID. Samples can be analyzed by these methods using direct injection, the headspace method (Method 5021) or the purge-and-trap method (Method 5030B and 5035). Groundwater samples should be determined using Method 5030B.

### 5.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

### 5.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

### 5.1.4 Sample Preparation

Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. Because of the large variability and complicated matrices of waste samples detection limits for this method may vary widely among samples.

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Method 5030 is a purge-and-trap method applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, groundwater, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 5035 is a purge-and-trap method applicable to nearly all types of soil samples, regardless of water content, including oily wastes, soils, and sediments.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

#### 5.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.
- c. Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.
- d. Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

#### 5.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

##### 5.1.6.1 Holding Times and Sample Preservation Criteria

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

- a. The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.
- b. No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

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- c. For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

#### 5.1.6.2 Holding Time and Sample Preservation Action

- a. Positive results in affected samples are generally qualified as estimated (J); non-detects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead.
- b. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); non-detects are generally considered to be unreliable and are qualified (UR).
- c. Results for which the holding time was grossly exceeded are biased low.

#### 5.1.6.3 Calibration Criteria

Verify the following:

- a. Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.
- b. In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity.
- c. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >20%. Circle these non-compliances on your working copies of these Forms. Spot check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.
- d. Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.
- e. Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the non-compliances on your working copies of these forms.

#### 5.1.6.4 Calibration Actions

- a. Generally, associated sample data are qualified as estimated (J), non-detects (UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.
- b. Generally, positive results for compounds for which %RSD or %D exceeds 20% or 15%, respectively, are qualified as estimated (J), non-detects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which reject non-detects if the %RSD or %D is excessive. Bias for these results cannot be determined.

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#### 5.1.6.5 Blank Contamination Criteria

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks.) Then repeat the process for contaminants occurring in the associated field quality control blanks.

#### 5.1.6.6 Blank Contamination Action

- a. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary amount protocols.
- b. Some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

#### 5.1.6.7 Surrogates Criteria

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any non-compliance on your working copies of these Forms.

#### 5.1.6.8 Surrogates Action

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided.

- a. For samples having a surrogate recovery <10%, positive results are qualified as estimated (J), non-detects are rejected (UR). These results are biased low.
- b. Samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); non-detects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- c. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Non-detects are not qualified based on high surrogate recoveries.

#### 5.1.6.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD)

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meet quality control limits. Circle outliers on the Form III or equivalent.

#### **5.1.6.10 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action**

- a. No action is generally taken on MS/MSD non-compliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results as estimated (J) and qualify non-detects as non-detected rejected (UR) in the unspiked sample.

#### **5.1.6.11 Field Duplicate Precision Criteria**

Compare the positive compound results with the results from the field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50%.

#### **5.1.6.12 Field Duplicate Precision Action**

Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); non-detects (UJ). Bias for these results cannot be determined.

#### **5.1.6.13 Laboratory Precision Criteria**

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds.

#### **5.1.6.14 Laboratory Precision Action**

Consider non-detects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

#### **5.1.6.15 Sample Result Verification Criteria**

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

#### **5.1.6.16 Sample Result Verification Action**

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support document section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory results within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory results is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

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### 5.1.6.17 Percent Solids Criteria

In some USEPA Regions a “Percent Solids” rule applies where each sample is analyzed to identify any samples that contain <30% solids.

### 5.1.6.18 Percent Solids Action

If any samples contains <30% solids, qualify positive and non-detected results as estimated (J) or non-detects (UJ) due to high moisture content of the sample.

### 5.1.7 Deliverable Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

## 5.2 Semivolatiles (SW8041/801A/8091/8310)

### 5.2.1 Applicability

Method 8041 is used to determine the concentration of the following phenolic compounds in groundwater, liquid, and solid matrices:

Phenol	4-Chloro-3-methylphenol
2-Chlorophenol	2,4-Dimethylphenol
2,4-Dichlorophenol	2-Nitrophenol
2,6-Dichlorophenol	4-Nitrophenol
Trichlorophenols	2,4-Dinitrophenol
Tetrachlorophenols	2-sec-Butyl-4,6-dinitrophenol (DNBP)
Pentachlorophenol	2-Cyclohexyl-4,6-dinitrophenol
Cresols (methyl phenols)	2-Methyl-4,6-dinitrophenol

Method 8061A is used to determine the concentration of the following phthalate esters in groundwater, liquid, and solid sample matrices:

Benzyl butyl phthalate  
Bis(2-ethylhexyl)phthalate  
Di-n-butyl phthalate  
Di-n-octyl phthalate  
Diethyl phthalate  
Dimethyl phthalate

Method 8091 is used to determine the concentration of the following nitroaromatic and cyclic ketone compounds in groundwater, liquid, and solid sample matrices:

Nitrobenzene  
Dinitrobenzene

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2,4-Dinitrotoluene  
2,6-Dinitrotoluene  
Isophorone  
Naphthoquinone

Method 8310 is used to determine the concentration of the following polynuclear aromatic hydrocarbons (PAHs) in liquid and solid sample matrices:

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(ghi)perylene	Phenanthrene
Benzo(k)fluoranthene	Pyrene

All of the above methods are gas chromatographic (GC), with the exception of Method 8310 which is a High Performance Liquid Chromatography (HPLC) technique, only (i.e., no mass spectrometer detector is employed). These methods use either an electron capture detector (ECD), a flame ionization detector (FID), an ultraviolet detector (UV), or a fluorescence detector.

### 5.2.2 Interferences

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source depending upon the waste being sampled. While general cleanup techniques such as Method 3530 are provided as part of these methods, unique samples may require additional cleanup.

If sample or matrix interferences occur, a secondary column may be employed in addition to the primary column so as to resolve any questionable compound results.

### 5.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

### 5.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using Method 3510 (separatory funnel extraction) or Method 3520 (continuous liquid-liquid extraction). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with

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methylene chloride using either Soxhlet Extraction (Method 3540) or Sonication (Method 3550) procedures.

### 5.2.5 Data Overview Prior to Validation

Before commencing validation the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

### 5.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

#### 5.2.6.1 Holding Times and Sample Preservation Criteria

Holding times are evaluated by reviewing COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

- a. For aqueous samples, use a 7-day maximum holding time until extraction.
- b. For soil samples, use a 14-day maximum holding time until extraction.
- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.

#### 5.2.6.2 Holding Times and Sample Preservation Action

- a. Positive results affected by non-compliance are qualified as estimated (J); non-detects (UJ). These results are considered to be biased low. Alternatively, the bias qualifiers L and UL may be used.
- b. Non-detects may be rejected (UR) when the sample was extracted (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high.

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Refer to the appropriate data validation protocol for specific guidance.

#### 5.2.6.3 Calibration Criteria

- a. Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.
- b. In general, either the correlation coefficient (R) of the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity.
- c. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >20%. Circle these non-compliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.
- d. Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with initial calibrations. Write the affected samples on your working copies of these forms.
- e. Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >15%; circle the non-compliances on your working copies of these forms.

#### 5.2.6.4 Calibration Action

- a. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.
- b. Positive results for compounds for which %RSD or %D exceeds 20% or 15%, respectively, are qualified as estimated (J); non-detects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which reject non-detects if the %RSD or %D is excessive. Bias for these results cannot be determined.

#### 5.2.6.5 Blank Contamination Criteria

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks.) Then repeat the process for contaminants occurring in the associated field quality control blanks.

#### 5.2.6.6 Blank Contamination Action

- a. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a

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common contaminant) are then set. The list of common contaminants may vary among protocols.

- b. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

#### **5.2.6.7 Surrogate Criteria**

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any non-compliance on your working copies of these Forms.

#### **5.2.6.8 Surrogate Action**

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided.

- a. For samples having a surrogate recovery <10%, positive results are qualified as estimated (J), non-detects are rejected (UR). These results are biased low.
- b. Samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); non-detects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance.
- c. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Non-detects are not qualified based on high surrogate recovery.

#### **5.2.6.9 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria**

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

#### **5.2.6.10 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action**

- a. Take no action based on MS/MSD non-compliances alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results in the unspiked sample as estimated (J) and qualify non-detects as rejected (UR).

#### **5.2.6.11 Field Duplicate Precision Criteria**

Compare the positive compound results with the results from the field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50%.

#### **5.2.6.12 Field Duplicate Precision Action**

Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); non-detects (UJ). Bias for these results cannot be determined.

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#### **5.2.6.13 Sample Result Verification Criteria**

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

#### **5.2.6.14 Sample Result Verification Action**

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation result in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

#### **5.2.6.15 Percent Solids Criteria**

Check the percent solids for each sample to identify any samples that contain <30% solids.

#### **5.2.6.16 Percent Solids Action**

- a. If any sample contains <30% solids, qualify positive and non-detected results as estimated (J); non-detects (UJ) due to high moisture content of the sample.
- b. If any sample contains <10% solids, qualify positive results as estimated (J); non-detects rejected (UR).

#### **5.2.6.17 Laboratory Precision Criteria**

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds.

#### **5.2.6.18 Laboratory Precision Action**

Consider non-detects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

#### **5.2.7 Deliverable Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation

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narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

### **5.3 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides (SW 8081A/8082/8141A/8151A)**

#### **5.3.1 Applicability**

Methods 8081A/8082 are used to determine the concentration of the following organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices:

Aldrin	Endrin
alpha-BHC	Endrin aldehyde
beta-BHC	Heptachlor
delta-BHC	Heptachlor epoxide
gamma-BHC (Lindane)	Methoxychlor
Chlordane	Toxaphene
4,4'-DDD	Aroclor-1016
4,4'-DDE	Aroclor-1221
4,4'-DDT	Aroclor-1232
Dieldrin	Aroclor-1242
Endosulfan I	Aroclor-1248
Endosulfan II	Aroclor-1254
Endosulfan sulfate	Aroclor-1260

Similarly, Method 8141A is used to determine the following pesticides in groundwater and waste samples:

Azinphos methyl	Fenthion
Bolstar (Sulprofos)	Merphos
Chlorpyrifos	Mevinphos
Coumaphos	Naled
Demeton-O	Parathion methyl
Demeton-S	Phorate
Diazinon	Ronnel
Dichlorvos	Stirophos (Tetrachlorvinphos)
Disulfoton	Tokuthion (Prothiofos)
Ethoprop	Trichloronate
Fensulfothion	

Note that when Method 8141A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique if mass spectroscopy is not employed.

Method 8151A is used to determine the following chlorinated acid herbicides in groundwater and waste samples:

2,4-D	Dichloroprop
2,4-DB	Dinoseb
2,4,5-T	MCPA
2,4,5-TP (Silvex)	MCPP

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Dalapon  
Dicamba

4-Nitrophenol  
Pentachlorophenol

Since these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A includes a hydrolysis step to convert the herbicide to the acid form prior to analysis. When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column; alternately, the compounds of interest can be confirmed by detection via a mass spectrometer.

All of the above Methods are Gas Chromatographic (GC) in which sample extracts are analyzed by direct injection. Methods 8081A and 8082 analyze for organochlorine pesticide compounds and PCBs via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used). Method 8141A analyzes for organophosphorous pesticide compounds via GC/FID (Flame Ionization Detector), and Method 8151A analyzes for chlorinated herbicide compounds via GC/ECD (alternately, a Microcoulometric Detector or Hall Electrolytic Conductivity Detector may be used).

### 5.3.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably, and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

### 5.3.3 General Laboratory Practices

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicate and laboratory duplicates should also be employed.

Note that herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, when performing Method 8151A, glassware and glass wool must be acid-rinsed and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

### 5.3.4 Sample Preparation

Prior to the use of Methods 8081, 8082, and 8141A, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid- liquid extractor (Method 3520). Solid samples are extracted with hexane:acetone (1:1) using either the Soxhlet extraction (Method 3540) or sonication (Method 3550) procedures.

Method 8151A provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetone and diethyl ether followed by esterification using diazomethane as a derivatizing agent.

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### 5.3.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- b. The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

### 5.3.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

#### 5.3.6.1 Holding Times and Sample Preservation Criteria

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

- a. For aqueous samples, use a 7-day maximum holding time until extraction.
- b. For soil samples, use a 14-day maximum holding time until extraction.
- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.

#### 5.3.6.2 Holding Times and Sample Preservation Action

- a. If holding times are exceeded, qualify positive results in affected samples as estimated (J); qualify non-detects (UJ). These results are usually assumed to be biased low unless prolonged storage causes a concentration increase, e.g., for degradation products which are also target analytes.
- b. If holding times are exceeded by a factor of more than two times the required time, qualify positive results as estimated (J); qualify non-detects rejected (UR). These exceedances are considered to be gross holding time exceedances.

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- c. If EPA Regional requirements apply, as in EPA Region III, apply the appropriate bias qualifiers as required; for example, detections and nondetects biased low (L) or (UL), respectively.

#### 5.3.6.3 Calibration Criteria

- a. Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form Vis or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels.
- b. Either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity.
- c. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >20%. Circle these non-compliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.
- d. Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.
- e. Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent differences (%Ds) 15%; circle the non-compliance on your working copies of these forms.
- f. Method 8081A requires analysis of a DDT/Endrin breakdown check standard. The DDT/Endrin Breakdown should not exceed 20%.

#### 5.3.6.4 Calibration Action

- a. Associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.
- b. Positive results for compounds for which %RSD or %D exceeds 20% or 15%, respectively, are qualified as estimated (J); non-detects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocols which reject non-detects if the %RSD or %D is excessive. Bias for these results cannot be determined.
- c. For Method 8081A if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE, and DDD following the in-last control standard until the next in-control standard (see analytical sequence). If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) non-detects for DDT in associated samples.
- d. If Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable standard. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) non-detect Endrin results.

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### 5.3.6.5 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed. Verify the following:

### 5.3.6.6 Blank Contamination Action

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

### 5.3.6.8 Surrogate Criteria

Evaluate surrogate recoveries by reviewing the laboratory data package Form II reports and the laboratory raw data.

- a. Verify that the recoveries are within the quality control ranges as given on the laboratory data package Form IIs; circle any non-compliance on your working copies of these Forms.
- b. Verify that the decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII are within +/- 0.10 for DCB and 0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peaks.

### 5.3.6.9 Surrogate Action

- a. No qualifications are made for surrogates which show zero recoveries because they were "diluted out."
- b. Positive results affected by low surrogate recovery are qualified as estimated (J) or the (L) bias qualifier is used when applicable; non-detects are qualified (UJ) or (UL), accordingly.
- c. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; non-detects are not qualified based on high surrogate recovery.
- d. Because the surrogate recovery limits for these fractions are advisory, generally no results are rejected.

### 5.3.6.10 Matrix Spike/Matrix Spike Duplicates

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

### 5.3.6.11 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Action

- a. Take no action based on MS/MSD non-compliances alone.

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- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results in the unspiked sample as estimated (J) and non-detects as rejected (UR).

#### 5.3.6.12 Field Duplicate Precision Criteria

- a. Check samples to determine if field duplicates were included in the data package.
- b. The Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <30%; for soil matrix results, <50% for sample results greater than the reporting limit.

#### 5.3.6.13 Field Duplicate Precision Action

- a. If positive results are greater than the reporting limit, qualify positive results for aqueous or soil media if the RPD exceeds 30% or 50% respectively. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J). Bias for these results cannot be determined.
- b. If one result is positive and the other is non-detected and the positive result is greater than 2 times the reporting limit, qualify positive and non-detected results as estimated (J) or (UJ), respectively.

#### 5.3.6.14 Sample Result Verification Criteria

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

#### 5.3.6.15 Sample Result Verification Action

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

#### 5.3.6.16 Percent Solids Criteria

Check the percent solids for each sample to identify any samples that contain <30% solids.

#### 5.3.6.17 Percent Solids Action

- a. If any sample contains <30% solids, qualify positive and non-detected results as estimated (J) or non-detectes (UJ), respectively, due to the high moisture content of the sample.

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- b. If any sample contains <10% solids, qualify positive results as estimated (J); qualify non-detected results as rejected (UR).

#### 5.3.6.18 Laboratory Precision Criteria

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds.

#### 5.3.6.19 Laboratory Precision Action

Consider non-detects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on this comparison.

#### 5.3.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

### 5.4 Explosives/Nitroaromatics/Nitroamines(SW 8330)

#### 5.4.1 Applicability

Method 8330 is used to determine the concentration of the following explosives, nitroaromatics, and nitroamines in water, soil, or sediment matrices:

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2,4-Dinitrotoluene (2,4-DNT)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	2,6-Dinitrotoluene (2,6-DNT)
1,3,5-Trinitrobenzene (1,3,5-TNB)	2-Nitrotoluene (2-NT)
1,3-Dinitrobenzene (1,2-DNB)	3-Nitrotoluene (3-NT)
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	4-Nitrotoluene (4-NT)
Nitrobenzene (NB)	Nitroguanidine
2,4,6-Trinitrotoluene (2,4,6-TNT)	Nitroglycerin
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	Pentaerythritol Tetranitrate (PETN)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	

The analysis of the compounds listed above is conducted by High Performance Liquid Chromatography equipped with a 254 nm Ultra Violet (UV) detector. This method is capable of determining part per billion (ppb) detection levels in water and soil matrices.

The method requires the use of both a primary (C-18 reverse phase) and a confirmation (CN reverse phase) column.

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#### 5.4.2 Interferences

The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

2,4-Dinitrotoluene and 2,6-dinitrotoluene may co-elute. High concentrations of one of the two isomers may cause interference of the other isomer. In instances where this is applicable, both isomers should be reported as one. Baseline resolution should be present for all compounds.

Decomposition of Tetryl occurs rapidly and when exposed to heat. Samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

#### 5.4.3 General Laboratory Practices

Method blanks and instrumentation blanks should be conducted to assess laboratory contamination.

Matrix spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field and laboratory duplicates may also be employed.

#### 5.4.4 Sample Preparation

Method 8330 provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetonitrile and a salting-out procedure for aqueous samples. Soil samples are air dried prior to preparation, thus percent moisture is not a consideration when calculating compound concentrations.

#### 5.4.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- a. If the appropriate number of samples are present in the data package.
- b. If each sample was correctly analyzed and identified for the specified parameters.
- c. The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and /or re-analyses, the validator should preview the data package contents to determine which analyses represent the best data quality.

Unless specifically directed by the client, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports ( including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

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#### **5.4.6 Technical Evaluation Summary**

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols, method requirements, and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this SOP is only intended as a general procedure for the data validation task.

Deficiencies, omissions, and/or other anomalies noted during the review require the data validator to contact the laboratory.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

##### **5.4.6.1 Holding Times and Sample Preservation Criteria**

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from the date of collection to the date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- a. For aqueous samples use a 7-day holding time until extractions.
- b. For solid samples use a 14-day holding time until extractions.
- c. For sample extracts use a holding time of 40 days from date of extraction to analysis.

##### **5.4.6.2 Holding Times and Sample Preservation Action**

- a. When the holding times criteria are not met, positive results in affected samples are generally qualified as estimated, (J); non-detected results, (UJ). These results are considered biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead.
- b. If the holding times are exceeded by a factor of two or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); non-detects are generally considered to be unreliable and are rejected, (R). These results are considered to be biased very low.

##### **5.4.6.3 Calibrations Criteria**

- a. Data pertaining to the initial calibration (i.e. evaluation check for linearity) is found on the data package Form Vis or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels. The initial calibration should consist of a minimum of five concentration levels for each compound of interest.
- b. Either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen the laboratory, the calibration curve should be checked for linearity.
- c. If the %RSD is used, determine which compounds have %RSD greater than 20%. Circle these non-compliances on working copies of calibration forms.

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- d. Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with each initial calibration by instrument. Write the affected samples on working copies of the appropriate continuing calibration forms. Spot-check (i.e. recalculate) a few of the %Ds to verify the laboratory's computation.
- e. A continuing calibration or daily calibration must be performed at the beginning, midpoint and end of the analytical sequence. The continuing calibration response factor for each analyte must be compared to the response factor of the initial calibration. The continuing calibration response factor must agree within 15% of the initial response factor.

#### **5.4.6.4 Calibrations Action**

- a. Associated sample data is qualified as estimated (J, UJ) if the calibration curve correlation coefficient is < 0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.
- b. Usually, associated data is qualified as estimated (J/UJ) if the calibration %RSD is >20%.
- c. Positive and non-detected results are qualified as estimated (J/UJ) if the Percent Difference (%D) is >15%.

#### **5.4.6.5 Blank Contamination Criteria**

A review of all method and instrument blanks (if provided) is conducted to evaluate laboratory contaminants. An additional review of all relevant field quality control blanks is also conducted. Contaminants, if present, are summarized and the maximum concentration of each contaminant is selected and used to establish blank action levels.

#### **5.4.6.6 Blank Contamination Action**

An action level of 5X the maximum amount of each contaminant is used to evaluate sample data. Blank action levels must consider the aliquot used for analysis and sample dilution. Positive results less than the action level are qualified as false positives. The manner in which the qualifiers are applied varies [i.e., use of (U) or (B); replacement by the Reporting Limit]. General regional guidance procedures dictate the most appropriate validation action qualification.

#### **5.4.6.7 Surrogates Criteria**

Surrogates are evaluated by reviewing the laboratory data package Form II or equivalent and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs. Circle any recoveries outside these limits on working copies.

#### **5.4.6.8 Surrogates Action**

- a. Positive results affected by low surrogate recoveries are estimated, (J) or (L), indicating low bias; non-detected results are qualified, (UJ) or (UL), accordingly.
- b. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J), or the bias qualifier (K), is used when applicable.
- c. Non-detected results are not qualified based upon high surrogate recoveries.

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- d. It should be noted that consideration of interferences may affect surrogate recoveries. If a trend of non-compliance is noted, an evaluation of sample chromatograms should be conducted when surrogate recoveries are noncompliant and a matrix effect is suspected.
- e. No qualifications are made for surrogates which have been diluted out.
- f. Positive results associated with surrogate recoveries <10% are qualified as estimated, (J) or biased low (L). Non-detected results associated with surrogate recoveries <10% are considered unreliable and are qualified rejected (R).

#### **5.4.6.9 Matrix Spike/Matrix Spike Duplicates (MS/MSD) Criteria**

Verify that matrix spike and matrix spike duplicate recoveries and Relative Percent Differences (RPD) meet quality control limits. Circle outliers on the Form III or equivalent.

#### **5.4.6.10 Matrix Spike/Matrix Spike Duplicates (MS/MSD) Action**

- a. Take no action based on MS/MSD non-compliance alone.
- b. If a matrix spike or matrix spike duplicate recovery is <10%, qualify positive results in the unspiked sample as estimated (J) and qualify non-detects as rejected (UR).

#### **5.4.6.11 Field Duplicate Criteria**

Compare positive compound results with MS/MSD analyses result for unspiked compounds. Generally, an RPD between field duplicate results for the aqueous matrix should be <30%; for soil matrix results <50%.

#### **5.4.6.12 Field Duplicate Action**

Qualification of the sample data is limited to specific field pair. Positive results for compounds showing imprecision are qualified as estimated (J); non-detects (UJ).

#### **5.4.6.13 Sample Result Verification Criteria**

Verify and record the quantitation of at least one compound per fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. The validator and laboratory quantitations must agree within 10%. If quantitation differences are significant, the laboratory must be contacted to investigate and resolve the discrepancy.

#### **5.4.6.14 Sample Result Verification Action**

- a. Perform a calculation verification of at least one analyte per fraction and include the re-calculation results in the support documentation section of the validation report. See Appendix A for calculation procedure.
- b. If the re-calculation does not agree with the laboratory result within 10%, contact the laboratory to determine whether the reviewer may have used incorrect information or if the laboratory result is incorrect and requires resubmission. A comment on the final outcome is required in the validation report along with the proper calculation verification.

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#### **5.4.6.15 Laboratory Precision Criteria**

Laboratory precision can be evaluated by comparing the unspiked samples results with MS/MSD analyses result for unspiked compounds.

#### **5.4.6.16 Laboratory Precision Action**

Consider non-detected results and results reported at concentrations less than the reporting limit to be in agreement. Use professional judgment in determining whether to qualify sample results based upon the comparison. The comparison may be presented in terms of a %RSD or an RPD.

#### **5.4.7 Deliverable Guidance**

In addition to any specific USEPA Regional requirements (i.e., data validation memorandum, data summary spreadsheets, USEPA Regional Worksheets), all laboratory data package quality summary forms, sample Form I reports method blank results and the Chain of Custody records must be included in the validation report.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the narrative is free of transcription and typographical errors before submitting all requested items for quality assurance review.

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## APPENDIX A, SAMPLE CALCULATIONS

Exhibit D Low/Medium Volatiles -- Section 11  
Data Analysis and Calculations (Con't)

11.2.1.2 Water

EQ. 7 Water Concentration Calculation

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{RRF}) (V_o)}$$

Where,

$A_x$  = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target compounds, internal standards, and DMCs are listed in Table 2.

$A_{is}$  = Area of the characteristic ion (EICP) for the internal standard. The target compounds are listed with their associated internal standards in Table 3.

$I_s$  = Amount of internal standard added, in ng.

$\overline{RRF}$  = Mean Relative Response Factor from the initial calibration.

$V_o$  = Total volume of water purged, in mL.

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e.,  $V_o$  above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5.0 mL with reagent water and purged,  $DF = 5.0 \text{ mL}/2.0 \text{ mL} = 2.5$ . If no dilution is performed,  $DF = 1.0$ .

11.2.1.3 Low-Level Soil/Sediment

EQ. 8 Low-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } (\mu\text{g/Kg}) \text{ (dry weight basis)} = \frac{(A_x) (I_s) (DF)}{(A_{is}) (\overline{RRF}) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$ , and DF are as given for water, Equation 7.

$\overline{RRF}$  = Mean Relative Response Factor from the heated purge of the initial calibration.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

$W_s$  = Weight of sample added to the purge tube, in g.

11.2.1.4 Medium-Level Soil/Sediment

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EQ. 9 Medium-Level Soil/Sediment Concentration Calculation

$$\text{Concentration } \mu\text{g/Kg (dry weight basis)} = \frac{(A_x) (I_s) (AV_t) (1000) (DF)}{(A_{is}) (\overline{RRF}) (V_a) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$  are as given for water, Equation 7.

$\overline{RRF}$  = Mean Relative Response Factor from the **ambient** temperature purge of the initial calibration.

$AV_t$  = Adjusted total volume of the methanol extract plus soil water in milliliters (mL) determined by:

$$AV_t = V_t + \{W_s - [W_s(D)]\}$$

Where  $V_t$  = total volume of methanol extract in milliliters (mL). This volume is typically 10 mL, even though only 1.0 mL is transferred to the vial in Section 10.1.5.5. The quantity derived from  $\{W_s - [W_s(D)]\}$  is the soil water volume and is expressed in mL.

$V_a$  = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100  $\mu\text{L}$ ), in microliters ( $\mu\text{L}$ ) added to reagent water for purging.

$W_s$  = Weight of soil/sediment extracted, in g.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

DF = Dilution Factor. The DF for analysis of soil/sediment samples for volatiles by the medium-level method is defined as:

$$\frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

11.2.1.5 For water, low-level and medium-level soil/sediment samples, xylenes are to be reported as "m,p-xylenes" and "o-xylene". Because m- and p-xylene isomers coelute, special attention must be given to the quantitation of the xylenes. In quantitating sample concentrations, be sure to use the correct corresponding Relative Response Factor (RRF) values.

NOTE: The area of each peak (i.e., the peaks for o-xylene and m,p-xylene) must appear on the complete quantitation report.

11.2.1.6 The stereoisomers, trans-1,2-dichloroethene, and cis-1,2-dichloroethene are to be reported separately.

11.2.1.7 Secondary ion quantitation is allowed **only** when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG Narrative. A secondary ion cannot be used unless an RRF is calculated using the secondary ion.

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D-42/LOW-MED VOA

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compound coelution, baseline noise, or matrix interferences. In these circumstances, the Contractor must perform a manual quantitation. Manual quantitations are performed by integrating the area of the quantitation ion of the compound. This integration shall only include the area attributable to the specific TCL compound. The area integrated shall not include baseline background noise. The area integrated shall not extend past the point where the sides of the peak intersect with the baseline noise. Manual integration is not to be used solely to meet Quality Control (QC) criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Any instance of manual integration must be documented in the SDG Narrative.

11.2.1.3 In all instances where the data system report has been edited or where manual integration or quantitation has been performed, the GC/MS Operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. The GC/MS Operator shall also mark each integrated area with the letter "M" on the quantitation report. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards, and DMCs.

11.2.1.4 The requirements listed in Sections 11.2.1.1 - 11.2.1.3 apply to all standards, samples, and blanks.

11.2.1.5 The Mean Relative Response Factor ( $\overline{RRF}$ ) from the initial calibration is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reason in the SDG Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a RRF is calculated using the secondary ion.

11.2.1.6 Calculate the concentration in the sample using the  $\overline{RRF}$  and Equations 5 and 6.

11.2.1.6.1 Water

EQ. 5 Concentration of Water Sample

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{RRF}) (V_o) (V_i)}$$

Where,

$A_x$  = Area of the characteristic ion for the compound to be measured.

$A_{is}$  = Area of the characteristic ion for the internal standard.

$I_s$  = Amount of internal standard injected in ng.

$V_o$  = Volume of water extracted in mL.

$V_i$  = Volume of extract injected in  $\mu\text{L}$ .

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$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$  (If GPC Cleanup is performed,  $V_t = V_{out}$ ).

$\overline{RRF}$  = Mean Relative Response Factor determined from the initial calibration standard.

$GPC = \frac{V_{in}}{V_{out}}$  = GPC factor. (If no GPC is performed,  $GPC = 1$ ).

$V_{in}$  = Volume of extract loaded onto GPC column.

$V_{out}$  = Volume of extract collected after GPC cleanup.

DF = Dilution Factor. The DF for analysis of water samples for semivolatiles by this method is defined as follows:

$$DF = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed,  $DF = 1.0$ .

11.2.1.6.2 Soil/Sediment

EQ. 6 Concentration of Soil/Sediment Sample

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x) (I_s) (V_t) (DF) (GPC)}{(A_{is}) (\overline{RRF}) (V_i) (W_s) (D)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$ ,  $V_{in}$ , and  $V_{out}$  are as given for water, above.

$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$   
(If no GPC Cleanup is performed, then  $V_t = 1000 \mu\text{L}$ .  
If GPC Cleanup is performed, then  $V_t = V_{out}$ ).

$V_i$  = Volume of the extract injected in  $\mu\text{L}$ .

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

$W_s$  = Weight of sample extracted in g.

$$GPC = \frac{V_{in}}{V_{out}} = \text{GPC Factor}$$

$\overline{RRF}$  = Mean Relative Response Factor determined from the initial calibration standard.

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DF = Dilution Factor. The DF for analysis of soil/sediment samples for semivolatiles by this method is defined as follows:

$$DF = \frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

A GPC factor of 2.0 is used to account for the amount of extract that is not recovered from the mandatory use of GPC cleanup. Concentrating the extract collected after GPC to 0.5 mL maintains the sensitivity of the soil/sediment method.

11.2.2 Non-Target Compound

An estimated concentration for non-target compounds tentatively identified shall be quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences shall be used. The equations for calculating concentration are the same as Equations 5 and 6. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compounds to be measured and the internal standard. An RRF of 1 is to be assumed. The resulting concentration shall be qualified as "J" (estimated, due to lack of a compound specific response factor), and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all TICs as well as those identified as unknowns.

11.2.3 CRQL Calculations

11.2.3.1 Water Samples

EQ. 7 Aqueous Adjusted CRQL

$$\text{Adjusted CRQL} = \text{Contract CRQL} \times \frac{(V_x)(V_t)(DF)}{(V_o)(V_c)}$$

Where,

$V_t$ , DF, and  $V_o$  are as given in Equation 5.

$V_x$  = Contract sample volume (1000 mL).

$V_c$  = Contract concentrated extract volume (1000  $\mu\text{L}$  if GPC is not performed. If GPC was performed, then  $V_c = V_{out}$ ).

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- 11.2.1.4 The Contractor must quantitate Toxaphene based on the Mean Calibration Factors (CFs) from the most recent initial calibration.
- 11.2.1.5 The chromatograms of all samples [including Laboratory Control Samples (LCSs), Matrix Spikes and Matrix Spike Duplicates (MS/MSDs)], standards, and required blanks must be reviewed by a qualified pesticide analyst before they are reported.
- 11.2.1.6 Calculate the sample concentration and on-column concentration of the single component pesticides and surrogates by using the following equations.
- 11.2.1.6.1 Water
- 11.2.1.6.1.1 EQ. 14 Concentration Calculation of Target Compounds in Water Samples

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_o) (V_i)}$$

Where,

$A_x$  = Response (peak area or height) of the compound to be measured.

$\overline{CF}$  = Mean Calibration Factor from the initial calibration (area/ng).

$V_{in}$  = Volume of extract loaded onto GPC column.

$V_{out}$  = Volume of extract collected after GPC cleanup.

$V_t$  = Volume of concentrated extract ( $\mu\text{L}$ ). (If GPC is not performed, then  $V_t = 10,000 \mu\text{L}$ . If GPC is performed, then  $V_t = V_{out}$ .)

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

$GPC = \frac{V_{in}}{V_{out}}$  = Gel Permeation Chromatography factor. (If no GPC is performed,  $GPC = 1.0$ )

$V_o$  = Volume of water extracted (mL). (NOTE: for instrument blanks and sulfur cleanup blanks, assume a 1,000 mL volume).

DF = Dilution Factor. The DF is defined as follows:

$$\text{DF} = \frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed,  $DF = 1.0$ .

The  $\overline{CF}$ s used in Equations 14 - 17 are those from the most recent initial calibration. If the CFs used to determine the linearity of the initial calibration were based on peak area, then the concentration of the analyte in the sample

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must be based on peak area. Similarly, if peak height was used to determine linearity, use peak height to determine the concentration in the sample.

11.2.1.6.1.2 EQ. 15 On-Column Concentration of Water Sample Extract

$$\text{On-Column Concentration (ng/}\mu\text{L)} = \frac{(A_x)}{(\overline{CF}) (V_i)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.6.2 Soil/Sediment

11.2.1.6.2.1 EQ. 16 Concentration of Target Compounds in Soil/Sediment Samples

$$\text{Concentration } \mu\text{g/Kg (Dry weight basis)} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_i) (W_s) (D)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_t$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

$W_s$  = Weight of sample extracted (g).

DF = Same as EQ. 14.

D = % dry weight or  $\frac{100 - \% \text{Moisture}}{100}$

GPC = Same as EQ. 14.

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11.2.1.6.2.2 EQ. 17 On-Column Concentration of Soil Sample Extract

$$\text{On-Column Concentration (ng/}\mu\text{L)} = \frac{(A_x)}{(\overline{CF})(V_i)}$$

Where,

$A_x$  = Same as EQ. 14.

$\overline{CF}$  = Same as EQ. 14.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.7 The lower of the two concentrations calculated for each single component pesticide is reported on Form I. In addition, the concentrations calculated for both the GC columns are reported on Form X, along with a Percent Difference (%Difference) comparing the two concentrations. The Percent Difference is calculated according to Equation 18.

EQ. 18 Percent Difference Between Concentrations on Both GC Columns

$$\%D = \frac{\text{Conc}_H - \text{Conc}_L}{\text{Conc}_L} \times 100$$

Where,

$\text{Conc}_H$  = The higher of the two concentrations for the target compound in question.

$\text{Conc}_L$  = The lower of the two concentrations for the target compound in question.

NOTE: Using this equation will result in Percent Difference values that are always positive.

11.2.1.8 The quantitation of Toxaphene must be accomplished by comparing the heights or the areas of each of the three or four major peaks of in the sample with the CF for the same peaks established during the initial calibration sequence. The concentration of Toxaphene is calculated by using Equations 14 and 16, where  $A_x$  is the area for each of the major peaks. The concentration of each peak is determined and then a mean concentration for the three or four major peaks is determined on each column.

11.2.1.9 The reporting requirement for Toxaphene is similar to that for the single component analytes, except that the lower mean concentration (from three or four peaks) is reported on Form I, and the two mean concentrations reported on Form X. The two mean concentrations are compared by calculating the Percent Difference using Equation 18.

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- 11.1.2.9 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above, and the concentration calculated from the GC/ECD analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form I with one of the laboratory-defined qualifiers ("X", "Y", or "Z"). In this instance, define the qualifier explicitly in the Sample Delivery Group (SDG) Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.
- 11.1.2.10 For GC/MS confirmation of Aroclors, spectra of three characteristic peaks are required for both the sample component and the reference standard.
- 11.1.2.11 The purpose of the GC/MS analysis for the Aroclors is to confirm the presence of chlorinated biphenyls in Aroclors. The GC/MS analytical results for the Aroclors shall not be used for quantitation and the GC/MS results shall not be reported on Form I and Form X. The exception noted in Section 11.1.2.9 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Calculations

11.2.1 Aroclor Concentrations

11.2.1.1 Water

11.2.1.1.1 EQ. 7 Concentration Calculation for Water Samples

$$\text{Concentration } \mu\text{g/L} = \frac{(A_x) (V_t) (DF) (GPC)}{(\overline{CF}) (V_o) (V_i)}$$

Where,

$A_x$  = Area or height of the peak for the compound to be measured.

$\overline{CF}$  = Mean Calibration Factor from the specific five-point calibration (area/ng).

$V_o$  = Volume of water extracted in mL (Note: for instrument and sulfur blanks assume a volume of 1000 mL).

$V_i$  = Volume of extract injected in  $\mu\text{L}$ . (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column).

$V_t$  = Volume of the concentrated extract in  $\mu\text{L}$ . (If GPC is not performed, then  $V_t = 10000 \mu\text{L}$ . If GPC is performed, then  $V_t = V_{out}$ ).

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DF = Dilution Factor. The DF for analysis of water samples by this method is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

$$\text{GPC} = \frac{V_{\text{in}}}{V_{\text{out}}} = \text{GPC factor. (If no GPC is performed, GPC} = 1.0).$$

$V_{\text{in}}$  = Volume of extract loaded onto GPC column.

$V_{\text{out}}$  = Volume of extract collected after GPC cleanup.

11.2.1.1.2 EQ. 8 On-Column Concentration of Water Sample Extract

$$\text{On-Column Concentration (ng}/\mu\text{L)} = \frac{(A_x)}{(\overline{\text{CF}}) (V_i)}$$

Where,

$A_x$  = Same as EQ. 7.

$\overline{\text{CF}}$  = Same as EQ. 7.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.1.2 Soil/Sediment

11.2.1.2.1 EQ. 9 Concentration Calculation for Soil Samples

$$\text{Concentration } \mu\text{g}/\text{Kg (Dry weight basis)} = \frac{(A_x) (V_t) (\text{DF}) (\text{GPC})}{(\overline{\text{CF}}) (V_i) (W_s) (D)}$$

Where,

$A_x$ ,  $V_t$ ,  $\overline{\text{CF}}$ , and GPC are as given for water in EQ 7.

$V_i$  = Volume of extract injected in  $\mu\text{L}$ . (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

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$$D = \frac{100 - \% \text{Moisture}}{100}$$

$W_s$  = Weight of sample extracted in g.

DF = Dilution Factor. The DF for analysis of soil/sediment samples by this method is defined as follows:

$$\frac{\mu\text{L most concentrated extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most concentrated extract used to make dilution}}$$

If no dilution is performed, DF = 1.0.

11.2.1.2.2 EQ. 10 On-Column Concentration of Soil Sample Extract

$$\text{On-Column Concentration (ng/\mu L)} = \frac{(A_x)}{(\overline{CF})(V_i)}$$

Where,

$A_x$  = Same as EQ. 7.

$\overline{CF}$  = Same as EQ. 7.

$V_i$  = Volume of extract injected ( $\mu\text{L}$ ). (If a single injection is made onto two columns, use  $\frac{1}{2}$  the volume in the syringe as the volume injected onto each column).

11.2.2 Target Compounds

The quantitation of Aroclors must be accomplished by comparing the heights or the areas of each of a minimum of 3 major peaks of the Aroclor in the sample with the  $\overline{CF}$  for the same peaks established during the specific five-point calibration. The concentration of multi-component analytes is calculated by using Equations 7 and 9, where  $A_x$  is the area for each of the major peaks of the Aroclor. The concentration of each peak is determined and then a mean concentration for a minimum of 3 major peaks is determined on each column.

11.2.2.1 Note that the  $\overline{CF}$ s used for the quantitation of Aroclors are the  $\overline{CF}$ s from the concentration of the specific five-point calibration.

11.2.2.2 The lower mean concentration (from a minimum of 3 peaks) is reported on Form I, and the two mean concentrations reported on Form X. The two mean concentrations are compared by calculating the Percent Difference (%Difference) using Equation 11.

## **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**

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
Teflon or Glass	All	FC 1131	Follow as written	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Extractable & Volatile Organics Petroleum Hydrocarbons		May omit acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit acid rinse
	Metals <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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FD 1000 Documentation Procedures

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

DEP-SOP-001/01  
FD 1000 Documentation Procedures

- 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)

## **FS 1000. GENERAL SAMPLING PROCEDURES**

See also the following Standard Operating Procedures:

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

### **FS 1001. Preliminary Activities**

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

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

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

#### 2. EQUIPMENT

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

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

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

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

#### 3. DEDICATED EQUIPMENT STORAGE

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

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

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

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

#### 4. SAMPLE CONTAINERS

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

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

## **FS 1002.** *Contamination Prevention and Sample Collection Order*

### 1. CONTAMINATION PREVENTION

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

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

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

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

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

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

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

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

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

### 2. SAMPLE COLLECTION ORDER

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

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

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

### 3. COMPOSITE SAMPLES

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

### **FS 1003.**     *Protective Gloves*

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

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

**FS 1004.**     *Container and Equipment Rinsing*

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

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

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

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

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

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

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

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

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

3.3. Holding Times for Automatic Samplers:

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

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

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

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

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

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

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

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

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

5. THERMAL PRESERVATION

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

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

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

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

**FS 1007.**     *Preventive and Routine Maintenance*

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

Equipment is properly maintained when:

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

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

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

2.1. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number or other unique identification.

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

2.3. Include the calendar date for the procedures performed.

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

2.5. Describe any malfunctions necessitating repair or service.

2.6. Retain vendor service records for all affected instruments.

2.7. Record the following for rented equipment:

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

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

**FS 1008.**     *Documentation and References*

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

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

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

**FS 1009.**     *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

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

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

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

## 2. LEGAL CHAIN OF CUSTODY (COC)

The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

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

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

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

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

2.1. Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times.

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

2.1.2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

2.1.3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

2.1.4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

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

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

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

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

#### 2.4. Shipping Samples under Legal COC

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

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

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

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

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

#### 2.5. Delivering Samples to the Laboratory

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

2.6. Chain of Custody Seals: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

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

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

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

### **FS 1010. *Health and Safety***

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

**FS 1011.**     *Hazardous Wastes*

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

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

**Appendix FS 1000**  
**Tables, Figures and Forms**

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

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

Construction Material <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 N/A	Purging	All analyte groups	None; <b>not recommended</b>
			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 <u>except</u> 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> <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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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

Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C <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 ( $1/100^{\text{th}}$  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 >

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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).

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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.

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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>⑤</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>⑤</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>⑧</sup>	Coring Device	5035 - Section 6.2.2.3 <sup>⑧</sup>	Glass Vial w/ PTFE-silicone Septum	6.1.3 <sup>⑧</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

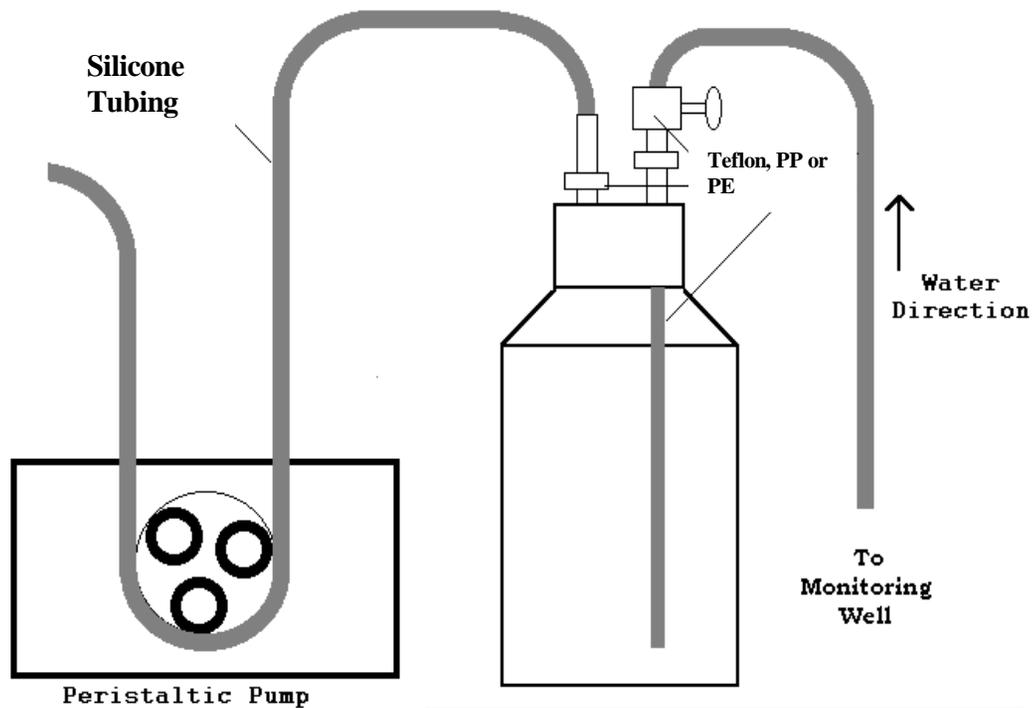
**NA – Not Applicable**

**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 1000. GENERAL SAMPLING PROCEDURES**

See also the following Standard Operating Procedures:

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

### **FS 1001. Preliminary Activities**

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

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

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

#### 2. EQUIPMENT

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

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

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

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

#### 3. DEDICATED EQUIPMENT STORAGE

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

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

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

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

#### 4. SAMPLE CONTAINERS

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

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

## **FS 1002.** *Contamination Prevention and Sample Collection Order*

### 1. CONTAMINATION PREVENTION

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

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

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

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

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

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

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

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

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

### 2. SAMPLE COLLECTION ORDER

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

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

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

### 3. COMPOSITE SAMPLES

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

### **FS 1003.**     *Protective Gloves*

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

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

**FS 1004.**     *Container and Equipment Rinsing*

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

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

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

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

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

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

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

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

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

3.3. Holding Times for Automatic Samplers:

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

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

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

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

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

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

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

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

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

5. THERMAL PRESERVATION

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

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

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

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

**FS 1007.**     *Preventive and Routine Maintenance*

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

Equipment is properly maintained when:

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

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

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

2.1. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number or other unique identification.

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

2.3. Include the calendar date for the procedures performed.

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

2.5. Describe any malfunctions necessitating repair or service.

2.6. Retain vendor service records for all affected instruments.

2.7. Record the following for rented equipment:

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

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

**FS 1008.**     *Documentation and References*

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

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

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

**FS 1009.**     *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

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

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

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

## 2. LEGAL CHAIN OF CUSTODY (COC)

The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

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

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

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

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

2.1. Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times.

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

2.1.2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

2.1.3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

2.1.4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

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

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

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

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

#### 2.4. Shipping Samples under Legal COC

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

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

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

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

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

#### 2.5. Delivering Samples to the Laboratory

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

2.6. Chain of Custody Seals: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

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

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

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

### **FS 1010. *Health and Safety***

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

**FS 1011.**     *Hazardous Wastes*

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

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

**Appendix FS 1000**  
**Tables, Figures and Forms**

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

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

Construction Material <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> <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

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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>
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

**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>
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C <sup>9</sup>	48 hours

Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C <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

**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>
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,

**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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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 >

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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).

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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**  
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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>⑤</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>⑤</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>⑤</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

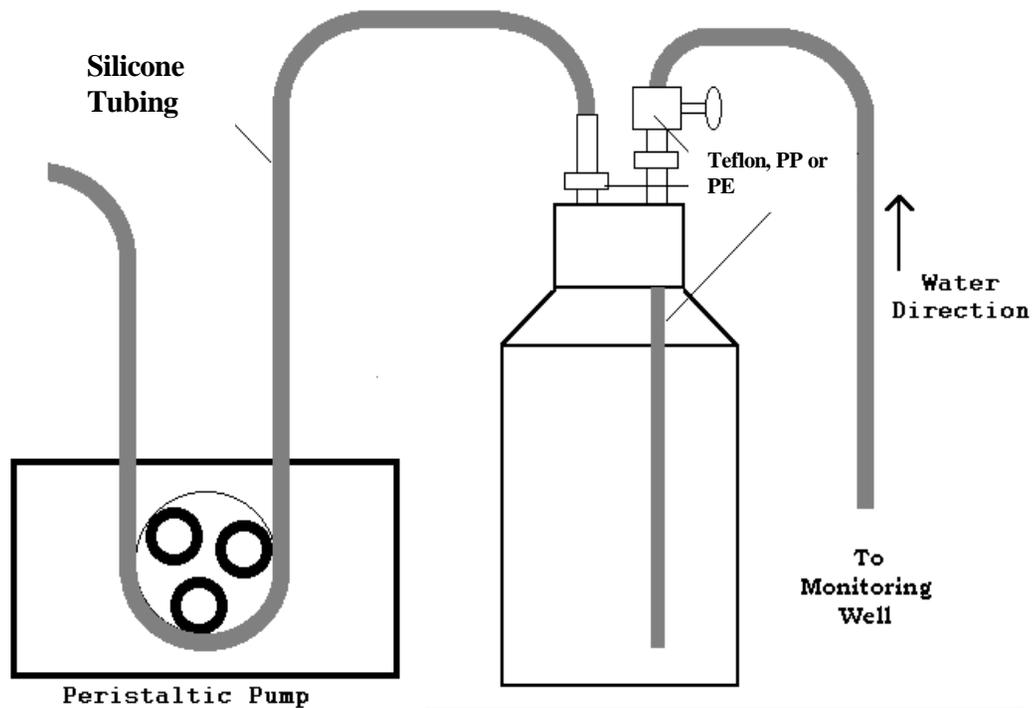
**NA – Not Applicable**

**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</b> (e.g., ISCO, Sigma)	
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 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**

Activity	Equipment Type
Well Purging	Variable speed centrifugal pump Variable speed submersible pump Variable speed bladder pump Variable speed peristaltic pump Bailer with lanyard: Not Recommended
Well Stabilization	pH meter DO meter Conductivity meter Thermometer/Thermistor Turbidimeter Flow-through cell Multi-function meters
Sample Collection	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Bailer with lanyard (See Table FS 2200-3)
Filtration	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Pressurized bailer 1.0 µm high capacity molded filter 0.45 µm high capacity molded filter
Groundwater Level	Electronic sensor Chalked tape

**Table FS 2200-2**  
**Dissolved Oxygen Saturation**

TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L
deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

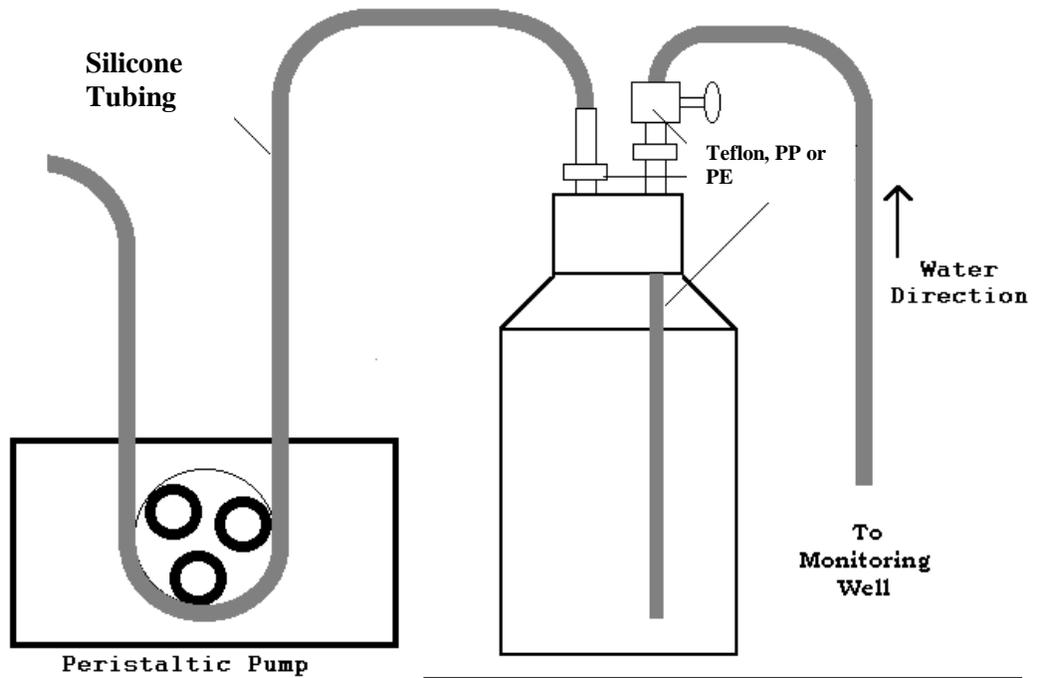
**Table FS 2200-3  
 Allowable Uses for Bailers**

• ANALYTE GROUP(S)	• PURGING (Not Recommended)	• SAMPLING	
	Use:	Use:	Not Recommended:
Volatile Organics Extractable Organics Radionuclides, including Radon Metals Volatile Sulfides	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If concentrations exceed action levels, the purpose is to monitor effective treatment, and the DEP program allows the use of bailers; or If specified by DEP permit, program, contract or order. or If operated by a skilled individual with documented training in proper techniques and using appropriate equipment. Field documentation must demonstrate that the procedure in FS 2221, section 2 was followed without deviation.	If concentrations are near or below the stated action levels; or If a critical decision (e.g., clean closure) will be made based on the data; or If data are to demonstrate compliance with a permit or order.
Petroleum Hydrocarbons (TRPH) & Oil & Grease	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	Only if allowed by permit, program, contract or order as samples should be collected into the container without intermediate devices.	Unless allowed by permit, program, contract or order.

DEP-SOP-001/01  
FS 2200 Groundwater Sampling

• ANALYTE GROUP(S)	• PURGING (Not Recommended)	• SAMPLING	
	Use:	Use:	Not Recommended:
Biologicals Inorganic Non-Metallics Aggregate Organics Microbiological Physical and Aggregate Properties	If allowed by permit, program, contract or order  or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If all analytes collected from the well can be collected with a bailer;  or If collected <u>after</u> collecting all analytes that require the use of a pump.	Before collecting any analytes that must be collected with a pump.
Ultra-Trace Metals	Never	Never	

**Figure 2200-1**  
**Pump and Trap for Extractable Organics**



The glass sample bottle must be threaded to use a reusable sampling cap lined and installed with fittings made of Teflon, polypropylene or polyethylene, similar to the design shown.

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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.

## **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.

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

calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

- 2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.
- 2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.
- 2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).
  - 2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.
  - 2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.
  - 2.6.3. Wait for stabilization, and read the pH value.
  - 2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.
3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.
4. DOCUMENTATION
  - 4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.
    - 4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.
      - 4.1.1.1. Document acceptable verification of any standard used after its expiration date.
    - 4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.
      - 4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.
      - 4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.
    - 4.1.3. Record the grade of standard or reagent used.
    - 4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.
      - 4.1.4.1. Record the date of preparation for all in-house formulations.
    - 4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).
  - 4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

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- 4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.
  - 4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.
    - 4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.
  - 4.2.3. Record the time and date of all initial calibrations and all calibration verifications.
  - 4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
  - 4.2.5. Record the name of the analyst(s) performing the calibration.
  - 4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
    - Type of standard or standard name (e.g., pH buffer)
    - Value of standard, including correct units (e.g., pH = 7.0 SU)
    - Link to information recorded according to section 4.1 above
  - 4.2.7. Retain manufacturers' instrument specifications.
  - 4.2.8. Document whether successful initial calibration occurred.
  - 4.2.9. Document whether each calibration verification passed or failed.
  - 4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
    - 4.2.10.1. Document date and time of any corrective action.
    - 4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
  - 4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 4.3. Record all field-testing measurement data, to include the following:
- Project name
  - Date and time of measurement or test (including time zone, if applicable)
  - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
  - Latitude and longitude of sampling source location (if required)
  - Analyte or parameter measured
  - Measurement or test sample value
  - Reporting units
  - Initials or name of analyst performing the measurement
  - Unique identification of the specific instrument unit(s) used for the test(s)

## FT 1200. Field Measurement of Specific Conductance (Conductivity)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

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

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

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

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

### 3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100  $\mu\text{mhos/cm}$  or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100  $\mu\text{mhos/cm}$  standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100  $\mu\text{mhos/cm}$ .

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100  $\mu\text{mhos/cm}$ , only one standard at 100  $\mu\text{mhos/cm}$  standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100  $\mu\text{mhos/cm}$  standard is 95 to 105  $\mu\text{mhos/cm}$ . If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using

the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}$$

Where: K = conductivity in  $\mu\text{mhos/cm}$  at 25°C

$K_m$  = measured conductivity in  $\mu\text{mhos/cm}$  at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}$$

Refer to SM2510B, 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  $\mu$ mhos/cm)
  - Link to information recorded according to section 5.1 above
- 5.2.7. Retain manufacturers' instrument specifications.
- 5.2.8. Document whether successful initial calibration occurred.
- 5.2.9. Document whether each calibration verification passed or failed.
- 5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
- 5.2.10.1. Document date and time of any corrective action.
- 5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 5.3. Record all field-testing measurement data, to include the following:
- Project name
  - Date and time of measurement or test (including time zone, if applicable)
  - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
  - Latitude and longitude of sampling source location (if required)
  - Analyte or parameter measured
  - Measurement or test sample value
  - Reporting units
  - Initials or name of analyst performing the measurement
  - Unique identification of the specific instrument unit(s) used for the test(s)

## FT 1300. Field Measurement of Salinity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

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

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

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

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

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

### 2. EQUIPMENT AND SUPPLIES

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

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

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

2.2. Standards:

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

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

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

2.3. Recordkeeping and Documentation Supplies:

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

3. CALIBRATION AND USE

3.1. Conductivity Method

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

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

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

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

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

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

3.1.1.6. Do not reuse standards for initial calibrations.

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

3.1.3. General Concerns for Conductivity Method

3.1.3.1. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

3.1.3.2. Thoroughly rinse the conductivity (salinity) sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample, or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

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

3.1.3.4. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the

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manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Density Method

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

$$D = a + bT^2$$

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

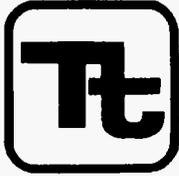
### 3.5. Measuring Turbidity in Samples

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.

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- 5.2.2.1. Record manufacturer name, model number, and identifying number (such as a serial number) for each instrument unit.
- 5.2.3. Record the time and date of all initial calibrations and all calibration verifications.
- 5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.
- 5.2.5. Record the name of the analyst(s) performing the calibration.
- 5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:
  - Type of standard or standard name (e.g., formazin)
  - Value of standard, including correct units (e.g., 20 NTU)
  - Link to information recorded according to section 5.1 above
- 5.2.7. Retain manufacturers' instrument specifications.
- 5.2.8. Document whether successful initial calibration occurred.
- 5.2.9. Document whether each calibration verification passed or failed.
- 5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.
  - 5.2.10.1. Document date and time of any corrective action.
  - 5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.
- 5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).
- 5.3. Record all field-testing measurement data, to include the following:
  - Project name
  - Date and time of measurement or test (including time zone, if applicable)
  - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
  - Latitude and longitude of sampling source location (if required)
  - Analyte or parameter measured
  - Measurement or test sample value
  - Reporting units
  - Initials or name of analyst performing the measurement
  - Unique identification of the specific instrument unit(s) used for the test(s)



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number GH-1.2	Page 1 of 9
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.

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ATTACHMENT A  
MONITORING WELL INSPECTION SHEET

**Monitoring Well Inspection Sheet**

Project Name: \_\_\_\_\_ Date: \_\_\_\_\_  
Location: \_\_\_\_\_ Time: \_\_\_\_\_  
Tidally Influenced: Y / N Personnel: \_\_\_\_\_

Field Measurements				
Well ID	PID Reading PPM	Depth to Water *	Total Depth *	Flush Mt./ Stick-up

Well Construction Details (Taken from construction logs)		
Total Depth *	Ground Elev.	Top/Btm Screen *

**Check List:**

Riser Pipe Material:
Riser Notched for Surveyors:
Well ID Tag In-place:
Well security:
Photo taken:

**Condition of Well:**

Protective Case:
Riser:
Well Pad:
Other:

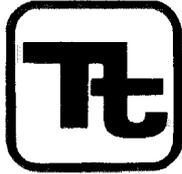
**Presence/Evidence of:**

Standing Water Around Well:
Existing Sampling Equipment:
Sediment build-up in Well Btm:

**Comments:**

\* = Measurements are from the top of the inner case to the nearest 0.01'





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 Smaller Than No. 200 Sieve Size		COARSE-GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	
GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION	GROUP SYMBOL	DESCRIPTION
OH	Highly organic silty clay with sand and siltstone fragments (see OH).	OH	Highly organic silty clay with sand and siltstone fragments (see OH).	OH	Highly organic silty clay with sand and siltstone fragments (see OH).
CI	Clayey inorganic silt with sand and siltstone fragments (see CI).	CI	Clayey inorganic silt with sand and siltstone fragments (see CI).	CI	Clayey inorganic silt with sand and siltstone fragments (see CI).
OL	Organic clayey silt with sand and siltstone fragments (see OL).	OL	Organic clayey silt with sand and siltstone fragments (see OL).	OL	Organic clayey silt with sand and siltstone fragments (see OL).
MI	Medium inorganic silt with sand and siltstone fragments (see MI).	MI	Medium inorganic silt with sand and siltstone fragments (see MI).	MI	Medium inorganic silt with sand and siltstone fragments (see MI).
ML	Low plasticity inorganic silt with sand and siltstone fragments (see ML).	ML	Low plasticity inorganic silt with sand and siltstone fragments (see ML).	ML	Low plasticity inorganic silt with sand and siltstone fragments (see ML).
CL	Clayey inorganic clay with sand and siltstone fragments (see CL).	CL	Clayey inorganic clay with sand and siltstone fragments (see CL).	CL	Clayey inorganic clay with sand and siltstone fragments (see CL).
OL	Organic clayey clay with sand and siltstone fragments (see OL).	OL	Organic clayey clay with sand and siltstone fragments (see OL).	OL	Organic clayey clay with sand and siltstone fragments (see OL).
SI	Silty inorganic sand with sand and siltstone fragments (see SI).	SI	Silty inorganic sand with sand and siltstone fragments (see SI).	SI	Silty inorganic sand with sand and siltstone fragments (see SI).
SM	Sandy inorganic silt with sand and siltstone fragments (see SM).	SM	Sandy inorganic silt with sand and siltstone fragments (see SM).	SM	Sandy inorganic silt with sand and siltstone fragments (see SM).
SC	Sandy clayey inorganic silt with sand and siltstone fragments (see SC).	SC	Sandy clayey inorganic silt with sand and siltstone fragments (see SC).	SC	Sandy clayey inorganic silt with sand and siltstone fragments (see SC).

CONSISTENCY OF GRANULAR SOILS		CONSISTENCY OF COHESIVE SOILS	
TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.	TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.
Very Loose	U < 1.5	Very Stiff	U > 10
Loose	1.5 < U < 2	Stiff	U > 10
Medium Loose	2 < U < 3	Medium Stiff	U > 10
Very Loose	U > 3	Very Stiff	U > 10
Overly Loose	U > 3	Overly Stiff	U > 10

ROCK HARDNESS (FROM CORE SAMPLES)		ROCK BROKENNESS	
TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.	TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.
Very Soft	U < 1.5	Very Soft	U < 1.5
Soft	1.5 < U < 2	Soft	1.5 < U < 2
Medium Soft	2 < U < 3	Medium Soft	2 < U < 3
Hard	U > 3	Hard	U > 3
Very Hard	U > 3	Very Hard	U > 3

SOIL SAMPLES - TYPES		SOIL SAMPLES - TYPES	
TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.	TERMINOLOGY	UNIFORMITY COEFFICIENT (U) AND CURVE NO.
1" Standard Sample	U < 1.5	1" Standard Sample	U < 1.5
2" Standard Sample	1.5 < U < 2	2" Standard Sample	1.5 < U < 2
Other Sample, Specific Methods	2 < U < 3	Other Sample, Specific Methods	2 < U < 3

1.5" Standard Sample  
 2" Standard Sample  
 Other Sample, Specific Methods

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### 5.2.1 USCS Classification

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

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

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

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

### 5.2.2 Color

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

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

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

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

### 5.2.3 Relative Density and Consistency

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

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

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

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

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

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

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

#### 5.2.4 Weight Percentages

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

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

## 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									
	22.0			22.0									
	23.0			23.0									
	24.0			24.0									
	25.0			25.0									
	26.0			26.0									
	27.0			27.0									
	28.0			28.0									
	29.0			29.0									
	30.0			30.0									

\* When rock coring, enter rock brokenness.  
 \*\* Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.  
 Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ± • 1-20Z  
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z Drilling Area  
NIX CORE IN BEDROCK RUN (1) = 25 min, RUN (2) = 15 min Background (ppm):   
 Converted to Well: Yes  No  Well I.D. #: MW-1

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- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
  - Particle shape - flat, elongated, or flat and elongated.
  - Maximum particle size or dimension.
  - Water level observations.
  - Reaction with HCl - none, weak, or strong.
- Additional comments:
  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

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- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.
- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

### 5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.
- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.
- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.
- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.
- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.
- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.
- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:
  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.
- Remarks at the bottom of Boring Log shall include:
  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.
- As a final check the boring log shall include the following:
  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.
  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

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### 5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

### 5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

### 6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

### 7.0 RECORDS

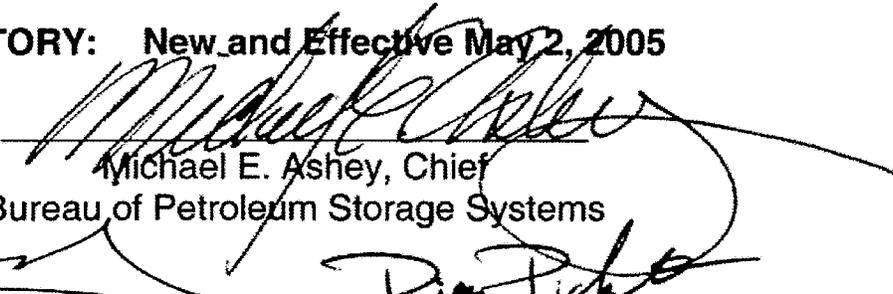
Originals of the boring logs shall be retained in the project files.

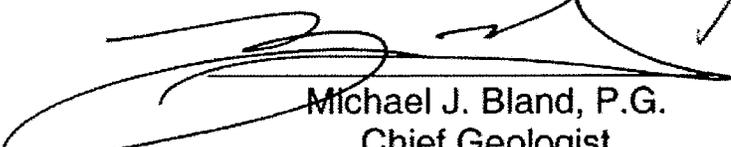
**DEPARTMENT OF ENVIRONMENTAL PROTECTION  
BUREAU OF PETROLEUM STORAGE SYSTEMS  
PETROLEUM CLEANUP PROGRAM**

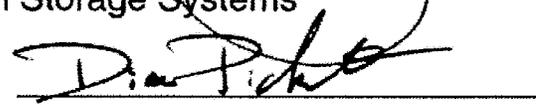
**STANDARD OPERATING PROCEDURES PCS-006**

**DESIGN, INSTALLATION, and PLACEMENT  
of  
MONITORING WELLS**

**HISTORY: New and Effective May 2, 2005**

  
Michael E. Ashe, Chief  
Bureau of Petroleum Storage Systems

  
Michael J. Bland, P.G.  
Chief Geologist

  
Diane D. Pickett, P.G.  
Assistant Chief Geologist

**Executive Summary**

The Florida Department of Environmental Protection (FDEP) Bureau of Petroleum Storage Systems (BPSS) presents this guidance document to clarify issues related to monitoring well design, installation, and placement in the Petroleum Cleanup Program (Program). Where practical, industry standard documents from the United States Environmental Protection Agency (USEPA) and the American Society for Testing and Materials (ASTM) have been referenced, with additions and exceptions noted where necessary to meet Program needs in a cost-effective manner. Any variance to this guidance must be approved by the BPSS in advance. Water Management District and/or Local Government rules, policies, and procedures must be followed if they are more stringent than those included in this guidance document. This guidance document replaces the August 1993 FDEP document entitled, Bureau of Waste Cleanup, "Monitoring Well Construction Specifications and Related Issues."

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**Attachments**

- Attachment A: Boring Log
- Attachment B: Drilling Methods - Advantages and Disadvantages
- Attachment C: Well Construction and Development Log

## **Design and Installation**

The BPSS requires monitoring well construction to be completed pursuant to the USEPA Region 4 guidance document entitled “Environmental Investigations Standard Operating Procedure and Quality Assurance Manual, Section 6: Design and Installation of Monitoring Wells” (<http://www.epa.gov/region4/sesd/eisopqam/eisopqam.pdf>), hereafter referred to as USEPA Region 4 guidance document, and microwell construction to be completed pursuant to the ASTM document entitled “Standard Guide for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers.” However, special exceptions and additions to this document are discussed below for a more comprehensive and practical guidance for monitoring well installations within the Florida BPSS program.

### **Well Permitting**

The various Water Management Districts (WMDs) in Florida differ in the permitting requirements and the permitting costs for monitoring wells and piezometers. A copy of all applicable Water Management District permits must be included in the reports that are submitted to the BPSS.

### **Utility Clearance**

A utility markout should be completed prior to any drilling activity. All boreholes need to be cleared to a minimum depth of four feet with a hand auger or post hole digger. The size of the borehole for the utility clearance should be at least as wide as the largest diameter auger or other equipment that will be placed within the borehole. Borings that are located in the vicinity of underground storage tanks (USTs), integral piping, or underground utility lines may require the borehole to be advanced to a deeper depth. Special caution should be taken for borings or wells located within 20 feet of power lines. If a monitoring well is needed within 20 feet of a power line, special precautions with the appropriate power company may be necessary (i.e., voltage of the line should be considered and if necessary the power company may need to shield the power lines to prevent arcing). Vacuum extraction techniques render the soil samples inadequate for OVA screening and laboratory analyses and should not be used for utility clearance for soil borings and wells unless prior approval is obtained from the BPSS. For a more detailed discussion on utility clearance for soil borings and wells placed near the petroleum storage and dispensing system, refer to the September 25, 2000 “Investigations Near Petroleum Storage Systems” (Exclusion Zone) memo prepared by the BPSS.

### **Drilling Methods**

There are several drilling methods that can be used to install acceptable monitoring and remediation wells in the Program. Presented in Attachment B is a list of drilling methods that are commonly used along with a brief description of the main advantages and disadvantages for each method. Additional information on the various drilling methods can be found in Section 6.3.1 of the USEPA Region 4 guidance document and Sections 7-9 of the ASTM D6724-01 guidance document.

Listed below are some comments to consider when using auger or rotary drilling methods:

#### Hand Bucket Auger:

If a hand bucket auger will be used to install a permanent monitoring well, prior approval from the BPSS should be obtained before the well is installed.

#### Hollow Stem Auger:

During hollow stem auger drilling, soil samples for lithology, laboratory analyses, and OVA screening must be collected from either split-spoon samplers or Shelby tubes, and not from off of the auger flights or from the pile of drill cuttings. The boring logs should clearly indicate the method that was used to obtain the soil samples and the amount of recovery of the sample. ASTM 5784-95 provides a useful and practical discussion on the use of split spoons.

#### Rotary Methods:

Section 6.3.4 of the USEPA Region 4 guidance document states that the best rotary method is water, followed by air, then mud. In Florida, the water or air rotary methods often are not appropriate due to the fine sand and silty sand lithologies that are commonly encountered, making the mud rotary method preferable. The water method may be practical in some clayey sediments because the clay formation could mix with the water and create a slurry with the appropriate consistency to carry the cuttings to the surface and keep the borehole open. However, some clays in the unsaturated zone may swell with the water causing swelling of the borehole walls and shrinking of the width of the borehole.

Although sonic and mud rotary are often used for the same difficult drilling conditions, there has been an increased use of sonic drilling during the past few years. The preference of sonic drilling over mud rotary drilling is partly due to issues associated with the drilling mud disposal costs and the problems associated with the retention of the drilling mud in the aquifer when mud rotary drilling is utilized.

### **Boring Log Requirements**

Boring logs are required for all borings/wells that are installed. Boring log requirements are discussed in the BPSS document "Soil Assessment and Sampling Methods" dated October 1, 2001 (the web link is: [http://www.dep.state.fl.us/waste/quick\\_topics/publications/pss/pcp/a-soil-MEMO.pdf](http://www.dep.state.fl.us/waste/quick_topics/publications/pss/pcp/a-soil-MEMO.pdf)). Since the October 1, 2001 guidance document was issued, the BPSS has permitted environmental consultants to use their own version of a boring log as long as all of the information that is specified in the October 1, 2001 guidance document is included on the boring logs. However, the BPSS has observed that the majority of the boring logs that are submitted do not contain all of the information that is required by the October 1, 2001 guidance document. The incomplete boring logs are due to the geologist not recording all of the required information in the field or because the boring log does not contain a column or a reminder to record the data for each sampled interval, such as moisture content. To address the issue of the incomplete boring logs, the BPSS has developed a

boring log form that contains all of the information that should be recorded during the completion of the soil borings and wells. The boring log is presented as Attachment A and the log is also available as an electronic copy at the Petroleum Cleanup Program's web site. The BPSS strongly recommends that the BPSS version of the boring log be used for all borings/wells that are installed. If the BPSS version of the boring log is not used, then all of the data that are included on the BPSS version of the log must be included on the environmental consultant's version of the boring log.

### **Monitoring Well Versus Piezometer**

There are many definitions in the literature for monitoring wells and piezometers. For the purposes of this guidance document, the BPSS will use the definitions provided in Subsections 62-770.200(31) and (41), Florida Administrative Code (F.A.C.) and listed below:

“Monitoring Well means a well constructed with a surface seal and a sand filter pack in accordance with accepted design practices in order to provide for the collection of representative groundwater samples for laboratory analyses. Such wells may also be used to detect the presence of free product or collect water-level elevation data to aid in determining the direction of groundwater flow.” A pre-packed microwell (direct-push well) is considered a type of monitoring well.

“Piezometer means a permanent or temporary well that may be designed and constructed without the surface sealing or sand filter pack requirements of a monitoring well. This type of well is primarily used to detect the presence of free product or collect water-level elevation data to aid in determining the direction of groundwater flow.”

Piezometers (often referred to as temporary wells) are generally installed and removed on the same day, but in some cases may remain on-site for longer periods. Examples of applications for piezometers include groundwater samples that are obtained for screening purposes with a direct-push rig or from a hand-augered boring, wells that are installed early in an assessment to obtain data on the groundwater flow direction to help in later placement of permanent monitoring wells, or wells that are installed and backfilled with native material and are used for free product measurements and delineation.

It is often helpful early in an assessment to obtain groundwater samples from piezometers for screening purposes, but the data that come from a piezometer without a sand pack should be used mostly for screening. There are cases where the laboratory data from a piezometer without a sand pack can be used for closure, but all the other information about the site (such as soil information, direction of groundwater flow, visual observations, and site history) must support the closure option and it must be demonstrated that the screen for the piezometer intersected the water table at the time that the sample was collected.

### **Well Construction**

Well construction specifications for monitoring wells installed with conventional drill rigs are outlined in Sections 6.4 through 6.6 of the USEPA Region 4 guidance document and for direct-push microwells in Section 7 of the ASTM D6725-01 document. The

construction of the wells should conform to the USEPA and ASTM documents, except as noted below in this guidance document.

### **A. Borehole Annular Space**

Because of the need for a 2-inch sand pack, the minimum borehole diameter for a 2-inch inside diameter (ID) monitoring well installed with a non-direct-push rig is six inches. Therefore, permanent wells installed with a conventional drilling rig require a minimum of two inches for the annulus located between the well screen and the wall of the borehole. For example, a 4 ¼-inch ID hollow-stem auger with 2-inch auger flighting will drill an 8 ¼-inch diameter borehole that will allow sufficient space for setting the well and placement of the minimum 2-inch sand pack. Hollow-stem augers with less than a 4 ¼-inch ID and/or less than 2-inch auger flighting do not allow sufficient room for proper well and sand pack placement. In some circumstances, such as when the aquifer is composed of sand that is similar to the sand pack material, exceptions to the minimum two-inch annular space for monitoring wells can be made. Variances to the two-inch annular space requirement must be approved by the BPSS prior to the installation of the wells.

The addition of water into the borehole during well installation is discouraged. However, in areas where flowing sands/silts may flow into the hollow-stem auger during well installation, caution should be taken to protect the integrity of the sand pack, such as filling the ID of the hollow-stem auger with water to compensate for the pressure of the flowing sands. If water must be introduced into the borehole, well development must be thorough and the well should not be sampled for seven days to ensure that the true formation waters are being sampled.

For 1-inch ID or greater microwells installed with a direct-push rig, there is no minimum annular space requirement because the microwells must be installed with pre-packed well screens. However, microwells are not well suited for installation in finer-grained materials such as silts and/or clays. If a combination direct-push/hollow-stem auger rig is used to install the monitoring well, then the two-inch minimum annular size requirement would apply if the hollow-stem auger method was used to install the well screen and riser.

### **B. Overdrilling the Borehole**

If a borehole was vertically overdrilled by five feet or greater, care should be taken to not allow a migration pathway through the undesired lower part of the borehole. Either backfill the bottom of the borehole with bentonite to the appropriate depth or properly abandon the borehole and then complete a borehole to the proper depth. Backfilling the borehole with sand is not permitted by the BPSS due to the potential for creating pathways for contamination migration.

### **C. Riser and Screen Size, Materials, and Connections**

As a general rule, the minimum ID casing size for most permanent monitoring wells must be two inches; however, exceptions can be made to install larger diameter casings, with prior BPSS approval. For permanent microwell installations, the minimum ID riser and screen size must be one inch.

Based on the fine sand lithologies that are commonly encountered in Florida, the preferable screen slot size for monitoring wells and microwells is 0.01 inch. If slot sizes greater than 0.01 inch are utilized, then a corresponding increase in the filter pack sand is required.

The preferred materials for monitoring well and microwell risers and screens are either Schedule 40 PVC or stainless steel. Variances to the Schedule 40 PVC or stainless steel requirement for monitoring wells or microwells must be approved by the BPSS prior to the installation of the well.

Sections of the screen and riser must be connected with flush threaded joints. If the sections can not be connected with threaded joints, then they must be mechanically fastened with slip caps that are permanently fastened with stainless steel screws. Glued or welded joints are not permitted.

#### **D. Filter Pack Material and Placement**

In general, the filter pack material for a 0.01 slot screen should be between 20 and 40 mesh silica sand. Refer to Section 6.6 of the USEPA Region 4 guidance document and the ASTM Standard D5092-90 for a discussion on determining the filter pack sizes for other screen slot sizes. A sieve analysis is not necessary for standard monitoring well installations in the Program to determine the size of the filter pack material. However, the collection of Shelby tube samples for sieve, hydrometer, bulk density, porosity, and permeability testing may provide useful information on site-specific hydrogeologic parameters and for the design of remediation wells if active remediation will be performed at the site.

The tremie method must be used for filter pack placement in monitoring wells. A cap must be placed on the end of the riser prior to filter pack placement to prevent sand from entering. Another acceptable method to set the filter pack is to pour the material directly into the annular space of the borehole provided that a PVC pipe is used as a tamping device to prevent bridging of the filter pack and that the amount of filter pack sand is continuously tagged during emplacement by the driller. In addition, the auger must be retrieved slowly to allow the filter pack to spread into the area of the well annulus occupied by the auger flights.

The filter pack sand should generally be placed to a depth of two feet above the well screen to allow settling of the filter pack during well development. For wells that have the top of the well screen beginning at depths of less than five feet, the amount of fine sand above the screen should be decreased in order to obtain a proper filter pack seal and surface seal for the well.

For microwells, pre-packed well screens must be utilized. U-pack wells and pouring, tamping, or using the tremie method for placing the filter pack sand into the direct-push borehole are not permitted by the BPSS.

#### **E. Filter Pack Seal Material and Placement**

The materials that are acceptable for use as a filter pack seal are bentonite and fine sand. Bentonite that is not properly hydrated will not form an effective seal for the filter pack. For this reason, the BPSS requires that fine sand be used as a filter pack seal for

“shallow” (water-table) wells and either bentonite or fine sand be used if the filter pack seal will be placed below the water table (e.g. vertical extent wells). Please note that if free product is present and the bentonite is in contact with the free product, then the bentonite may not hydrate properly.

The tremie method or the tamping method (see filter pack installation) must be used to install the bentonite or fine sand. The thickness of the fine sand or the hydrated thickness of the bentonite should be a minimum of two feet. If fine sand is used, the thickness should be a minimum of two feet. For wells that have the top of the well screen beginning at depths of less than five feet, the amount of the filter pack seal must be proportionately decreased in order to maintain a proper amount of sand pack above the well screen and grout surface seal above the filter pack seal.

Elevated pH levels may indicate that the filter pack seal was improperly installed. Special attention must be paid to the pH levels while purging the well for the first time to determine if the bentonite or fine sand provided an effective seal for the grout surface seal.

#### **F. Surface Seal Material and Placement**

The purpose of the surface seal is to prevent surface water run-off from migrating down the outside of the well casing or the borehole. For a list of materials that can be used for grouting the annular space, please consult Section 6.4.5 of the USEPA Region 4 guidance document. The annular space of the monitoring wells and microwells must also be grouted according to WMD regulations.

#### **G. Well Surface Completion**

Monitoring wells that are completed above grade may or may not require a protective steel casing, depending on whether or not they are located in a heavy traffic area. The amount of stickup of the well should not exceed 2.5 feet and the exact height above the land surface should be proportional to how difficult it is to find the well (such as in vegetation or non-consolidated material), whether the well will be a traffic concern, and whether surface water levels may rise and reach the well location.

Flush-to-grade wells must be installed with a manhole set in a concrete well pad that has a minimum size of two feet wide by two feet long. For manholes that are larger in diameter than 10 inches, the well pad size should be proportionately increased. A minimum of one inch of the finished pad must be below grade to prevent washing and undermining by soil erosion. It may not be necessary to install a two-foot by two-foot concrete pad if the well is installed in a concrete paved surface. The concrete well pad must be domed slightly to prevent surface water runoff from entering the manhole, but should not be excessively domed as to create a tripping hazard. If the well is completed flush-to-grade and is installed in a heavy traffic area, a bolt-down manhole cover must be installed.

Requirements for Monitoring Well Caps: Well caps serve two main functions: to prevent liquids and gases from entering or escaping from the well, and to discourage tampering with the well. The BPSS requires certain construction standards to ensure that the integrity of the well cap is sufficient, and to either prevent unauthorized access to the well or leave evidence of tampering if it has occurred. Below is a description of the

requirements that must be inherent in the design and materials used in the manufacturing of a monitoring well cap. If any of the requirements listed below are not followed, or if the cap is faulty, then the cap will need to be replaced at the environmental contractor's or responsible party's expense.

1. The cap must not contain any corrosive metals that have the potential to leach from the cap into the monitoring well. If metal parts exist that have the potential to leach into the well, they must be composed of stainless steel or other non-corrosive metal types.
2. The well plug should be liquid/air tight to prevent liquids and gases from entering or escaping from the well. A quality well plug must be able to withstand hydrostatic pressures/vacuums from the formation and should be constructed of materials that will not degrade in an unreasonable time frame at a typical site and compromise the seal. The requirement that the cap be liquid/pressure tight applies to all wells, including small-diameter direct-push microwells. Water-tight manhole covers should not substitute for the requirement that the plug be liquid/pressure tight.
3. The well casing must be sealed along the inside diameter (ID) or outside diameter (OD) of the casing with a one-piece liquid/air tight rubber gasket. The gasket material must have a broad range of chemical resistance and absorption properties. Such recommended materials include Santoprene®, Geolast® or Buna-nitrile. Use of the proper gasket materials guards against well intrusion and will withstand repeated use.
4. The caps must be tamper resistant (capable of locking and with a thick plastic tie used to lock the cap). The plastic tie must be of sufficient thickness so that it can only be removed by cutting the tie. Each time the well is accessed, the plastic tie should be cut and replaced. Padlocks should not be used to lock the cap. The well designation must be secured to the plastic tie or permanently affixed to the well pad using a steel tag or etched into the concrete pad. Locking well caps must be inserted and tightened according to the manufacturer's instructions. The cap must not be pulled out of the well casing using the plastic tie. The purpose of a locking well cap is to keep intruders out and/or to inform the owner/operator of the well that unauthorized access has occurred. If a well cap has been maliciously and forcefully wrenched from its casing, it must have characteristics in its construction **not** allowing it to be stepped, stomped, jammed, or hammered back into the well casing.
5. The expansion caps need to be inspected each time the well is accessed and replaced if faulty (such as if the expansion cap is stripped or the seal on the cap is worn).

## **H. Well Construction Details**

Well construction details need to be included in the report that is submitted to the BPSS. A copy of the required Well Construction and Development Log that needs to be provided for each well that is installed is provided in Attachment C. An electronic copy of the Well Construction and Development Log is also available at the Program's web site. This form should not be modified and must be entirely filled out.

## I. Well Development

For a discussion of various well development techniques, refer to Section 6.8 of the USEPA Region 4 guidance document.

Development is required for all permanent water-table and vertical extent monitoring wells, and microwells. Adding water to a monitoring well for development purposes is not permitted. The wells should be developed after a sufficient amount of water has recharged into the well. The amount of the water removed from the well during the development process should be a function of one or more of the following:

1. If possible, adding water to a monitoring well during the drilling process should be avoided or minimized. If water must be added during the drilling of the borehole, then at least five times as much water as was added to the borehole during the drilling process must be removed during the development of the well.
2. For wells installed using the mud-rotary method, a more aggressive development method must be utilized for an extended period of time to remove the drilling mud from the filter pack and formation.
3. The well must be developed until the water is clear of any visible suspended particulate matter.
4. If all of the particulate matter in the development water can not be removed after a sufficient amount of time, then the development can be stopped at the discretion of the consultant, but must be documented in the comment section of the Well Construction and Development Log. Every effort should be made to properly develop a well, because proper development will produce a more representative groundwater sample from the formation and will reduce the amount of time that it takes for parameters to stabilize during future groundwater sampling activities for the well.

Generally, when developing a permanent monitoring well, the aquifer should be stressed in order to remove the fine-grained particles that are trapped in the filter pack and adhered to the borehole wall. Therefore, avoid developing 2-inch and greater diameter wells with pumps that pump at a low flow rate, such as peristaltic pumps.

The purpose of developing piezometers is often to just remove the bulk of the suspended particulate matter located in the well casing or direct-push screen point sampler prior to obtaining a groundwater sample for lab analyses. Therefore, when developing a piezometer that will be sampled immediately after development, minimize the amount of development time by not developing for a prolonged period to obtain a perfectly clear sample and avoid purging more than five water well volumes from the piezometer.

The monitoring of pH, temperature, specific conductance, dissolved oxygen, and turbidity readings are not necessary while developing a well. Recording the estimated amount of drawdown, development pumping rate, and whether the well purges dry during development can, however, provide useful information that can be used at a later date for estimating the initial pumping rate during groundwater purging activities prior to

sampling, and provides information concerning the hydraulic characteristics of the aquifer.

For all permanent monitoring wells and microwells, a well development log is required to be submitted to BPSS with the reports. A copy of the required Well Construction and Development Log that needs to be provided for each well that is developed is provided in Attachment C.

Piezometers can be sampled immediately after development. Monitoring wells should not be sampled until at least 24 hours after development. In cases where water was added to the monitoring well during the drilling activities or air development methods were used, groundwater sampling should not be performed for at least seven days after the well development activities were completed. Waiting seven days ensures that the chemistry of the groundwater has not been altered by the addition of water or air during development of the well.

#### **J. Soil Boring and Well Abandonment**

Soil borings need to be properly abandoned so that they do not serve as a preferential conduit for contamination. Monitoring wells need to be abandoned after the site rehabilitation activities are completed or after the well is determined to be no longer useful so that the well can not be used in the future for unauthorized access. The soil borings and wells need to be abandoned in accordance with the WMD requirements, and any exceptions to the grouting requirements must be approved by the appropriate WMD. For information on soil boring and well abandonment, consult the appropriate WMD and refer to Section 6.9 of the USEPA Region 4 guidance document and ASTM Standard D5299-92.

With the increased use of direct-push rigs, the BPSS has observed in recent years that there have been a greater number of soil borings that have been completed to assess the soil and to determine the proper placement for permanent monitoring wells. In many instances the soil borings have been backfilled with the native soils or with sand. If the material that is placed in a soil boring is more permeable than the surrounding formation, the backfilled material in the former soil boring will serve as a preferential pathway for contamination.

For sites that have a very shallow water table, where soils are contaminated down to the water table, or where the lithology consists of homogeneous sand, grouting the soil boring is not always necessary. As a general guideline, the BPSS presents the following rules to follow when determining if a soil boring needs to be properly abandoned with grout:

1. If the soil boring is suspected to have penetrated a perched zone or a confining or semi-confining interval.
2. If the site has a deep water table and the lithology consists of heterogeneous sands and clays.
3. If soil contamination is identified, but the groundwater is not suspected of being impacted by the contamination.

4. If a direct-push rig is utilized to collect groundwater samples or lithologic data from below the water table to characterize the lithology and to determine the vertical extent of the plume.

Whenever there is doubt as to whether the soil boring will serve as a preferential pathway for the migration of contaminants, the soil boring must be grouted. The method used for abandonment of the soil boring must be included on the boring log.

Boreholes and wells must be grouted from the bottom up by means of a tremie pipe. In addition, dry bentonite pellets must not be placed into the borehole due to the difficulty in hydrating the bentonite pellets to form an effective seal.

When abandoning monitoring wells, the well pad and manhole must be removed but the riser and the screen must be left in place unless there is evidence that the monitoring well was improperly constructed. The riser and screen must be filled with grout, the manhole should be removed, and the area that was formerly occupied by the well pad should be repaired so that the concrete or asphalt is flush with the existing grade. A copy of each well abandonment form must be included in the report to the BPSS.

#### **K. Investigation-Derived Waste (IDW)**

During the well installation event, the environmental consultant should make a determination as to whether the development water and the drill cuttings should be drummed for off-site disposal.

1. Generally, development water should be pumped slowly to a paved surface to allow for evaporation, as long as surface runoff to an unpaved area does not occur. The exceptions to pumping the development water to a paved surface are:
  - a. If free product is detected at the water table interface during the installation of the well.
  - b. Dissolved contaminants are suspected in the groundwater and there are no paved surfaces at the site. Discharging the development water in the unpaved source area would contaminate soil at the surface that has not been impacted by the petroleum discharge or would contaminate a different aquifer zone (e.g., a perched zone).
2. In the past, soil was drummed during well installation or soil boring activities if there was no place on-site to spread it or if it was “excessively” contaminated (>500 ppm on the OVA for gasoline and >50 ppm for diesel). The implementation of soil cleanup target levels (SCTLs) makes spreading the soil based solely on OVA screening results inappropriate. If soil analytical data have not been collected at a site, then the drill cuttings should be drummed, sampled for laboratory analyses, and if the results indicate that SCTLs are exceeded, disposed of off-site. Soil cuttings should be placed in drums during the initial field event at a site, then future decisions about soil disposal will be based on laboratory data, with OVA correlations when possible. Under some circumstances, such as at small sites, ultimate disposal decisions could be based on practicality

and cost-effectiveness as well as laboratory data, as some cuttings might not exceed SCTLs but cannot be spread on the source property due to space limitations. All soil should be screened with an OVA during the advancement of the boring for the well unless an exception has been agreed to by the BPSS.

Please note that pursuant to Standard Operating Procedures PCS-005, the BPSS will require that calibration records be kept for all field equipment used (including the OVA) pursuant to Section FT 1000 of the Groundwater Sampling SOP. A calibration log (Form FD 9000-8) is included in Section FT 1000 of the SOP for recording the calibration data in the field. The BPSS will require Form FD 9000-8 to be filled out for all field equipment used in the gathering of data. Documentation of field calibration events must also be documented in the field log.

## **Placement**

### **Strategy for Well Placement**

The strategy for placement of monitoring wells can vary based on many factors at a site, including: the depth to the water table, the presence of perched intervals, access issues on-site and off-site, lithology and the presence of multiple aquifers, the type and amount of the discharge(s), and the location of soil contamination. A direct-push rig can often be used to obtain groundwater screening samples for determining the optimal locations for permanent water-table wells and the screen interval of vertical extent wells.

Alternatively, an assessment investigation can also be initiated by installing permanent wells with a conventional drilling rig to determine the magnitude of petroleum contamination in the source area.

For any assessment investigation where contamination is detected, there needs to be a minimum of three water-table monitoring wells or piezometers installed in order to determine the direction of groundwater flow. The wells should not be installed in a line, but should be installed in a triangular manner so that the direction of groundwater flow can be determined with greater certainty.

As a general rule, additional monitoring wells that are installed outside of the source areas should be spaced apart by 30 to 50 feet. The larger spacing should be employed farther away from the source areas for sites that have very high dissolved concentrations and for sites that have very deep water tables. The spacing of wells may be decreased or increased depending on access issues, delineating the extent of free product, and the dissolved contaminant levels that are detected.

Vertical extent wells should be installed next to or slightly downgradient (within five feet) of the most contaminated source wells. For sites that have multiple source areas, a vertical extent well should be installed at each source area where the dissolved concentrations exceed the natural attenuation default source concentrations that are specified in Table V of Chapter 62-777, F.A.C.

### **Determining the Need for Additional Monitoring Wells**

As an assessment progresses, the need for and placement of additional monitoring wells must be evaluated. The following are general guidelines to be followed:

1. If the dissolved contamination in the groundwater exceeds the natural attenuation default concentrations for source wells, then the plume should be “chased” horizontally and vertically. Exceptions to this are:
  - a. Physical barriers in the way (e.g., buildings and very wide roads).
  - b. The existence of a confining clay layer greater than 5-10’ thick vertically that is laterally continuous across the area of the plume. The existence of the confining clay layer must be agreed upon by the consultant’s Professional Geologist of record and the BPSS’s Professional Geologist of

record. (NOTE: For the purposes of this guidance document the terms confining, semi-confining, and retarding are subjective. Geotechnical testing is rarely completed on the soils before monitoring well installation, so the lithology description is generally based on a field interpretation.)

- c. The resampling of the vertical extent well indicates that the dissolved contamination detected in the first groundwater sampling event in excess of the natural attenuation criteria does not represent the actual dissolved concentrations in the formation. Frequently, the first groundwater sampling event that is conducted for a vertical extent well may erroneously indicate that the results are in excess of the natural attenuation criteria. This “false positive” occurs as the result of the “dragging down” of soil or groundwater contamination from the shallow zone during the installation of the single or double-cased vertical extent well. Before any additional vertical extent wells are installed to define the base or lateral extent of the plume in the deep zone, the vertical extent well should be evaluated for resampling to confirm the first groundwater analytical results.
2. If groundwater contamination exceeds the groundwater SCTLs but is less than the natural attenuation default source concentrations, an evaluation of whether to “chase” the plume should be made on a case-by-case basis as outlined below:
    - a. Temporary groundwater screening samples obtained using direct-push rigs are generally from very short-screened intervals located at the water table. Groundwater samples obtained from permanent monitoring wells that have a longer screened interval and are installed at the same locations of the direct-push groundwater screening locations often display a decrease or non-detect contaminant levels.
    - b. Where groundwater cleanup target levels are only slightly exceeded in monitoring wells, it may be possible to use isoconcentration contour lines to estimate the extent of the plume on the source property, and avoid installing additional permanent monitoring wells to locate the exact spot where the dissolved contamination is at or below the cleanup target levels. Isoconcentration contour maps must be submitted with multiple contour lines, which demonstrate the concentration gradient across the plume. Logarithmic contour lines (e.g., 10,000 ug/L, 1,000 ug/L, 100 ug/L, 10 ug/L, and 1 ug/L) are preferred to show plume characteristics for sites with high concentrations.
    - c. If potable well(s) or other receptors are reasonably close, especially in the downgradient direction, additional monitoring well installations to better define the plume are warranted.
    - d. If the site will undergo natural attenuation monitoring and there are no off-site access issues, there must be a monitoring well demonstrating a clean downgradient point of compliance.

If contamination in the vertical extent well is confirmed at levels in excess of the natural attenuation default source concentrations, then additional vertical delineation of the contamination consisting of a deeper vertical extent well may be necessary and the horizontal extent of the "intermediate" contamination may also be required. The horizontal extent of intermediate contamination is often necessary when a downward vertical gradient is present suggesting that the intermediate impacted aquifer may have a different lithology, hydraulic gradient, and/or flow direction. Determining the horizontal extent of the intermediate zone contamination also might be necessary if the intermediate zone lithology appears to be more transmissive than the shallow zone materials. If the lithology is consistent through the depth of concern, additional horizontal delineation may not be necessary at that depth if the distribution of the contaminants of concern can be inferred from the concentrations in the shallower zone.

### **Length and Placement of the Well Screen for Water-Table (“Shallow”) Monitoring Wells**

For water-table (“shallow”) monitoring wells, 10 to 15 feet of screen should be used to bracket the water table during the seasonal fluctuations of the water table. Screen lengths of greater than 15 feet are inappropriate for the following two reasons:

1. A longer screen length can allow transfer of contaminants from the upper parts of the screen to the lower parts.
2. A longer screen length is often considered necessary to cover an apparently large fluctuation of the water table (for example 10 to 20 feet). In most cases, this apparent fluctuation is not an accurate representation of the true water table, but is usually due to a perched or semi-perched zone above the regional water table that tends to accumulate water for days, weeks, or even months after rain events. The perched or semi-perched zones can be seasonal and only support a water column when the influx of rain is greater than the perched or semi-perched zone’s ability to allow it to percolate through or flow around the perched or semi-perched zone. Small amounts of finer material in the soils will impede the downward gravity flow of water from rain events. After rain events, or in the rainy season, water can be slow to infiltrate through the finer materials. If a site appears to have a water table fluctuation greater than 10 to 15 feet, rather than utilizing a screened interval of 20 feet or greater, it would be more appropriate to place two monitoring wells with shorter screened intervals clustered next to each other. The upper perched or semi-perched zone should be screened with one well, and the deeper zone should be screened by another well and the two screened intervals should not overlap in a manner that allows the migration of contaminants from one screened interval to the next. This way, contamination in the shallower and deeper zones will not mix and a more accurate estimate of where the depth of the contamination is can be made.

If a piezometer is being utilized for assessment of free product, the screened interval must include the capillary fringe area.

During an initial site assessment event, it can be difficult to determine the depth of the water table, especially when fine materials are present. Over the past few years, with the increased use of initial screening events utilizing direct-push rigs for quick screening of

water levels and water quality, the BPSS has seen an increased number of monitoring wells set to improper depths. It is apparent that sufficient time and appropriate methods are not being utilized for the water table to stabilize before the correct depth to water is being noted. An appropriate effort must be made to determine the proper depth to water to allow for proper placement of the well screens. The well screens should be placed to intersect the current water table at the time of the drilling event and should also intersect the estimated water table during seasonal fluctuations. As a standard practice, the top of the well screen should not always be arbitrarily placed two feet above the water table that was observed at the time of drilling. The effects of seasonal fluctuations have to be considered when determining the placement of the screen. The current depth to water and the seasonal fluctuation range observed at the site can be determined by the following:

- a. Reviewing historical water-level data obtained from existing wells located at the site or from any nearby sites prior to the drilling event.
- b. Considering the seasonal effects of precipitation and whether the site area is in a prolonged drought condition or abnormally wet period.
- c. Gauging any existing wells on-site before installing any additional wells.
- d. When taking water levels during direct-push screening events, if fines such as silts or clays are present, allow sufficient time for the water table to stabilize in the borehole before the final depth to water is determined. In some cases (such as in areas with finer-grained materials) this will require setting a length of well screen in the borehole and allowing the water table to stabilize as other work is being completed on the site. It is not uncommon for the water table to take hours to stabilize. If it doesn't recharge within 24 hours, the existence of a different water-bearing zone should be considered.
- e. Observations about moisture content (e.g., dry, damp or moist, wet, saturated) as a boring is advanced should be noted and recorded on the boring log. Changes in the moisture content will help determine the existence and depth of the true water table or any perched zones that may be present. The different categories of water content will have varying observed properties depending on the lithology present and the drilling method used. A saturated sand will have different properties than a saturated clay. Field staff must be trained and sufficiently experienced to interpret the visual observations of moisture content and lithologies to help determine the depth at which the soils are saturated. Moisture content observations during the advancement of the borehole are also very helpful in determining the actual depth to the water table in aquifers that are under confining or semi-confining conditions.
- f. Using observed spikes in the OVA concentrations obtained from above the current water table within the smear zone and away from the immediate source area to determine the approximate seasonal high water table.
- g. If installing wells over multiple days for wells that have good to moderate recharge, gauge wells that were previously installed (e.g., the day before or earlier the same day) to determine the depth to water before drilling any additional wells.

If the depth to water is less than two feet, then the monitoring well screen should begin at a depth of two feet below land surface in order to allow proper placement of the filter pack above the screen, filter pack seal, and the surface seal. If free product is present at a site that has a water table of less than two feet, then piezometers should be installed in the area of the free product and screened across the water table (completed above grade when possible).

### **Criteria for Determining if Vertical Extent Wells are Necessary**

Vertical extent wells are installed to help determine the vertical extent of groundwater contamination and to help describe the lithology of the treatment area when active remediation is being considered. Vertical extent wells should generally be installed when:

1. The petroleum contaminants of concern in the water-table wells are greater than the natural attenuation default source concentrations that are specified in Table V of Chapter 62-777, F.A.C.
2. Private or public supply wells are located in close proximity to the plume or supply well impacts have been documented.
3. In areas with a known or suspected high vertical (downward) hydraulic gradient or where there is sufficient reason to believe the plume is being pulled downward, such as in karstic formations (as determined by the consultant's Professional Geologist of record and agreed upon by the BPSS's Professional Geologist of record).

### **Length and Placement of the Well Screen for Vertical Extent Wells**

The maximum screen length for a vertical extent well should be five feet (or more if the screened portion of the aquifer is slow to recharge). Due to the short screened interval, proper placement of the screen within a permeable interval is preferable. If it is suspected that the vertical extent well will not yield a sufficient water sample due to the confining or retarding nature of the soils, then consideration should be given to collecting a soil sample for lab analyses at the base of the vertical extent well. This result will provide additional information along with the OVA data as to the vertical extent of the plume.

The placement of the five-foot screened interval should be based on the following:

1. If a confining (retarding) interval is identified during the drilling activities, the top of the filter pack for the vertical extent well screen should be at least two feet below the confining interval.
2. If an OVA/FID is utilized for screening purposes, the OVA data should be collected from above and below the water table. A sharp decrease in OVA readings can be used as an indicator in determining the depth of the plume and the placement of the screened interval.
3. If a direct-push rig is utilized, groundwater samples can be obtained at approximately 10 to 15 foot intervals with the screen point sampler to

determine the base of the plume. If a retarding unit is encountered during the drilling activities, then this method should not be used below the retarding interval unless surface casing is driven into the retarding unit.

4. If the lithological data indicate that a retarding unit is not present and the OVA data can not be used to approximate the base of the plume, then the top of the screen for the vertical extent well should be installed between 10 to 20 feet below the bottom of the screens of the water-table wells.
5. The top of the filter pack for the vertical extent well screen should be separated from the surface casing by a minimum of 3 feet. This should help prevent any cross contamination resulting from the installation of the surface casing.

### **Placement of the Surface Casing for Double- or Triple-Cased Vertical Extent Wells**

The following criteria must be considered to determine whether a permanent or temporary surface casing should be used during installation of a vertical extent well:

1. To minimize the potential for drag-down of the surficial contamination during well installation. The installation of the surface casing should prevent the vertical extent well from acting as a conduit for downward migration of the groundwater contamination if free product or “significant” dissolved contamination exists in the upper section of the impacted aquifer (significant may be defined as greater than 2000 ug/L total BTEX or PAHs).
2. If evidence exists of a perched water table, or when drilling in a heterogeneous stratified lithology (especially when there is concern that the well sand pack annulus might breach a retarding clay unit).

Triple casing may be considered at sites with very high contamination levels (free product), significantly contaminated intermediate zones, and/or large vertical hydraulic gradients. On a site-by-site basis, an evaluation should be made as to the need for additional casing to segregate lithologic/hydrologic units by separate casings.

Temporary casings used with direct-push and sonic drilling methods are acceptable, but the consultant should describe the backfilling techniques to ensure formation bridging is minimized.

The diameter of the surface casing should allow for the proper placement of the well casing and the 2-inch minimum annular space for the filter pack. Generally, the surface casing should extend at least a few feet past the bottom of the water-table well (or the adjacent impacted vertical extent well). The main goal is to isolate the vertical extent well from the source contamination at the screened interval of the adjacent impacted well. A reduction in OVA levels or lab data in soils retrieved from below the surficial aquifer can provide an indication as to the bottom of the source/smear zone. But the lithology should also be a determining factor when setting the depth of the surface casing. When screening a vertical extent well below a clay lens, set the casing within the upper two feet of the lens (if possible). Even if it is known that this is not a continuous unit, the clay will provide a good seat for the surface casing, which will minimize the drag-down of contamination.

If additional vertical extent wells are needed to determine the lateral extent of contamination observed in the initial vertical extent well, a review of the shallow aquifer contamination levels should be performed to determine if a surface casing is needed for any of the additional wells. This is especially important when the "intermediate" contaminant plume extends beyond the extent of the shallow contamination (as defined by the water-table monitoring wells). The evaluation of whether to double-case any additional vertical extent wells should be based on the following three factors:

- Lithology.
- Zone of contamination.
- Existence of a confining/retarding unit.

## References Cited

ASTM, Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (D5092-90, Reapproved 1995).

ASTM, Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities (D5299-92).

ASTM, Standard Guide for Use of Hollow-Stem Augers for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices (D5784-95, Reapproved 2000).

ASTM, Standard Guide for Installation of Direct Push Ground Water Monitoring Wells (D6724-01).

ASTM, Standard Guide for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers (D6725-01).

USEPA, Environmental Investigations Standard Operating Procedure and Quality Assurance Manual, Section 6: Design and Installation of Monitoring Wells, November 2001.

## Other References

ASTM, Standard Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices (D5783-95, Reapproved 2000).

ASTM, Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations (D6001-96).

FDEP, Interested Parties – 2, Technical Review Section, Bureau of Waste Cleanup (September 29, 1992).

FDEP, Interested Parties – 4, Technical Review Section, Bureau of Waste Cleanup (November 20, 1995).

ATTACHMENT A

**Boring Log**

# BORING LOG

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Boring/Well Number:		Permit Number:		FDEP Facility Identification Number:	
Site Name:		Borehole Start Date:	Borehole Start Time:	<input type="checkbox"/> AM <input type="checkbox"/> PM	
		End Date:	End Time:	<input type="checkbox"/> AM <input type="checkbox"/> PM	
Environmental Contractor:		Geologist's Name:		Environmental Technician's Name:	
Drilling Company:		Pavement Thickness (inches):	Borehole Diameter (inches):		Borehole Depth (feet):
Drilling Method:	Apparent Borehole DTW (in feet from soil moisture content):	Measured Well DTW (in feet after water recharges in well):	OVA (list model and check type):		
			<input type="checkbox"/> FID	<input type="checkbox"/> PID	
Disposition of Drill Cuttings [check method(s)]: <input type="checkbox"/> Drum <input type="checkbox"/> Spread <input type="checkbox"/> Backfill <input type="checkbox"/> Stockpile <input type="checkbox"/> Other					
<i>(describe if other or multiple items are checked):</i>					
Borehole Completion (check one): <input type="checkbox"/> Well <input type="checkbox"/> Grout <input type="checkbox"/> Bentonite <input type="checkbox"/> Backfill <input type="checkbox"/> Other (describe)					

Sample Type	Sample Depth Interval (feet)	Sample Recovery (inches)	SPT Blows (per six inches)	Unfiltered OVA	Filtered OVA	Net OVA	Depth (feet)	Sample Description (include grain size based on USCS, odors, staining, and other remarks)	USCS Symbol	Moisture Content	Lab Soil and Groundwater Samples (list sample number and depth or temporary screen interval)
							1				
							2				
							3				
							4				
							5				
							6				
							7				
							8				
							9				
							10				
							11				
							12				

Sample Type Codes: PH = Post Hole; HA = Hand Auger; SS = Split Spoon; ST = Shelby Tube; DP = Direct Push; SC = Sonic Core; DC = Drill Cuttings  
 Moisture Content Codes: D = Dry; M = Moist; W = Wet; S = Saturated

# BORING LOG

Boring/Well Number:		FDEP Facility Identification Number:				Site Name:		Borehole Start Date:			
								End Date:			
Sample Type	Sample Depth Interval (feet)	Sample Recovery (inches)	SPT Blows (per six inches)	Unfiltered OVA	Filtered OVA	Net OVA	Depth (feet)	Sample Description (include grain size based on USCS, odors, staining, and other remarks)	USCS Symbol	Moisture Content	Lab Soil and Groundwater Samples (list sample number and depth or temporary screen interval)
							13				
							14				
							15				
							16				
							17				
							18				
							19				
							20				
							21				
							22				
							23				
							24				
							25				
							26				
							27				
							28				
							29				
							30				

Sample Type Codes: PH = Post Hole; HA = Hand Auger; SS = Split Spoon; ST = Shelby Tube; DP = Direct Push; SC = Sonic Core; DC = Drill Cuttings  
 Moisture Content Codes: D = Dry; M = Moist; W = Wet; S = Saturated

# BORING LOG

Page \_\_\_\_\_ of \_\_\_\_\_

Boring/Well Number:		FDEP Facility Identification Number:			Site Name:			Borehole Start Date:			
								End Date:			
Sample Type	Sample Depth Interval (feet)	Sample Recovery (inches)	SPT Blows (per six inches)	Unfiltered OVA	Filtered OVA	Net OVA	Depth (feet)	Sample Description (include grain size based on USCS, odors, staining, and other remarks)	USCS Symbol	Moisture Content	Lab Soil and Groundwater Samples (list sample number and depth or temporary screen interval)

Sample Type Codes: **PH** = Post Hole; **HA** = Hand Auger; **SS** = Split Spoon; **ST** = Shelby Tube; **DP** = Direct Push; **SC** = Sonic Core; **DC** = Drill Cuttings  
 Moisture Content Codes: **D** = Dry; **M** = Moist; **W** = Wet; **S** = Saturated

ATTACHMENT B

**Drilling Methods Advantages and Disadvantages**

<b>Drilling Method</b>	<b>Main Advantages</b>	<b>Main Disadvantages</b>
<b>Hand Bucket Auger</b>	<ul style="list-style-type: none"> <li>• Inexpensive method for soil assessment and the installation of temporary and/or permanent monitoring wells with shallow water tables.</li> <li>• Appropriate for areas of difficult access (e.g., swamps, heavy woods, medians, or overhead utilities).</li> </ul>	<ul style="list-style-type: none"> <li>• Labor intensive.</li> <li>• Caving may occur during the well installation.</li> <li>• Limited to shallow depths (generally less than 10 feet).</li> <li>• Requires a core drill in areas covered with asphalt and/or concrete.</li> </ul>
<b>Hollow Stem Auger</b>	<ul style="list-style-type: none"> <li>• Appropriate in unconsolidated material to shallow and intermediate depths.</li> <li>• Facilitates the collection of soil samples from split-spoon samplers for lithologic and OVA data.</li> </ul>	<ul style="list-style-type: none"> <li>• Sample recovery is less reliable than soil samples collected with Direct-Push Rigs.</li> <li>• Access limitations with small sites, swamps, heavy woods, overhead utilities, and under canopies.</li> </ul>
<b>Rotary Method: Water, Air, Mud</b>	<ul style="list-style-type: none"> <li>• Appropriate in unconsolidated or consolidated material to shallow and deep depths.</li> <li>• Appropriate for many difficult drilling conditions that other methods can not properly overcome.</li> </ul>	<ul style="list-style-type: none"> <li>• Soil sampling is time consuming due to time required to remove the rotary drill bit and pipe from the borehole for sample collection.</li> <li>• Access limitations with small sites, swamps, heavy woods, overhead utilities, and under canopies.</li> <li>• Difficult to install wells in limestone with large voids due to circulation loss.</li> <li>• Increased well development time due to time required to remove the drilling mud.</li> <li>• Time consuming to install multiple cased wells.</li> <li>• Generates a large amount of investigation derived waste (IDW).</li> </ul>

<p><b>Sonic</b></p>	<ul style="list-style-type: none"> <li>• Appropriate in unconsolidated and consolidated material to intermediate and deep depths.</li> <li>• Preferred for installing monitoring wells in hard limestone with large voids.</li> <li>• Less IDW generated compared to mud rotary.</li> <li>• Temporary override casing instead of permanent surface casing, so reduced drilling time for multiple-cased wells.</li> <li>• Samples may be collected in the drill casings for lithologic characterization.</li> <li>• Appropriate for many difficult drilling conditions that other methods can not properly overcome.</li> </ul>	<ul style="list-style-type: none"> <li>• Cost prohibitive for most shallow monitoring wells.</li> <li>• Often more expensive than mud rotary.</li> <li>• Access limitations with small sites, swamps, heavy woods, overhead utilities, and under canopies (access limitations are less of a concern if a mini-sonic rig is used).</li> <li>• Samples from consolidated material are often pulverized.</li> <li>• Poor sample recovery from unconsolidated fine sand lithologies lacking clay.</li> </ul>
<p><b>Direct-Push (DPT)</b></p>	<ul style="list-style-type: none"> <li>• Appropriate in unconsolidated material to shallow depths.</li> <li>• Often good recovery of soil samples for lithology, laboratory analyses, and OVA data.</li> <li>• Sample liners prevent loss of volatiles.</li> <li>• Groundwater samples are collected using a screen point sampler or by placing PVC screen into the borehole.</li> <li>• Variety of DPT rig sizes to accommodate limited access.</li> <li>• Minimal IDW.</li> </ul>	<ul style="list-style-type: none"> <li>• Difficult in areas of swelling clays, well-sorted sands, or in former UST areas backfilled with pea gravel.</li> <li>• Not appropriate for consolidated material such as limestone or areas that are backfilled with concrete and asphalt fill.</li> <li>• Generally limited to shallow depths.</li> </ul>

ATTACHMENT C

**Well Construction  
and  
Development Log**

## WELL CONSTRUCTION AND DEVELOPMENT LOG

<b>WELL CONSTRUCTION DATA</b>					
Well Number:		Site Name:		FDEP Facility I.D. Number:	
Well Location and Type (check appropriate boxes): <input type="checkbox"/> On-Site <input type="checkbox"/> Right-of-Way <input type="checkbox"/> Off-Site Private Property <input type="checkbox"/> Above Grade (AG) <input type="checkbox"/> Flush-to-Grade		Well Purpose: <input type="checkbox"/> Perched Monitoring <input type="checkbox"/> Shallow (Water-Table) Monitoring <input type="checkbox"/> Intermediate or Deep Monitoring <input type="checkbox"/> Remediation or Other (describe)		Well Install Date(s):	
If AG, list feet of riser above land surface:				Surface Casing Install Method:	
Borehole Depth (feet):	Well Depth (feet):	Borehole Diameter (inches):	Manhole Diameter (inches):	Well Pad Size: _____ feet by _____ feet	
Riser Diameter and Material:		Riser/Screen Connections: <input type="checkbox"/> Flush-Threaded <input type="checkbox"/> Other (describe)		Riser Length: _____ feet from _____ feet to _____ feet	
Screen Diameter and Material:		Screen Slot Size:		Screen Length: _____ feet from _____ feet to _____ feet	
1 <sup>st</sup> Surface Casing Material: also check: <input type="checkbox"/> Permanent <input type="checkbox"/> Temporary		1 <sup>st</sup> Surface Casing I.D. (inches):		1 <sup>st</sup> Surface Casing Length: _____ feet from <u>  0  </u> feet to _____ feet	
2 <sup>nd</sup> Surface Casing Material: also check: <input type="checkbox"/> Permanent <input type="checkbox"/> Temporary		2 <sup>nd</sup> Surface Casing I.D. (inches):		2 <sup>nd</sup> Surface Casing Length: _____ feet from <u>  0  </u> feet to _____ feet	
3 <sup>rd</sup> Surface Casing Material: also check: <input type="checkbox"/> Permanent <input type="checkbox"/> Temporary		3 <sup>rd</sup> Surface Casing I.D. (inches):		3 <sup>rd</sup> Surface Casing Length: _____ feet from <u>  0  </u> feet to _____ feet	
Filter Pack Material and Size:		Prepacked Filter Around Screen (check one): <input type="checkbox"/> Yes <input type="checkbox"/> No		Filter Pack Length: _____ feet from _____ feet to _____ feet	
Filter Pack Seal Material and Size:				Filter Pack Seal Length: _____ feet from _____ feet to _____ feet	
Surface Seal Material:				Surface Seal Length: _____ feet from _____ feet to _____ feet	

<b>WELL DEVELOPMENT DATA</b>			
Well Development Date:		Well Development Method (check one): <input type="checkbox"/> Surge/Pump <input type="checkbox"/> Pump <input type="checkbox"/> Compressed Air <input type="checkbox"/> Other (describe)	
Development Pump Type (check): <input type="checkbox"/> Submersible <input type="checkbox"/> Other (describe)		Depth to Groundwater (before developing in feet):	
Pumping Rate (gallons per minute):		Maximum Drawdown of Groundwater During Development (feet):	
Pumping Condition (check one): <input type="checkbox"/> Continuous <input type="checkbox"/> Intermittent		Well Purged Dry (check one): <input type="checkbox"/> Yes <input type="checkbox"/> No	
Total Development Water Removed (gallons):		Development Duration (minutes):	
Development Water Drummed (check one): <input type="checkbox"/> Yes <input type="checkbox"/> No			
Water Appearance (color and odor) At Start of Development:		Water Appearance (color and odor) At End of Development:	

<b>WELL CONSTRUCTION OR DEVELOPMENT REMARKS</b>



**TETRA TECH**

# STANDARD OPERATING PROCEDURES

Number SA-1.1	Page 1 of 34
Effective Date 04/07/2008	Revision 7
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject  
GROUNDWATER SAMPLE ACQUISITION AND  
ONSITE WATER QUALITY TESTING

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## 1.0 PURPOSE

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

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

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

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

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

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

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

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

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

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

#### 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

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

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

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

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

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

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

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

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

## 5.0 HEALTH AND SAFETY

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

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

Methods of avoiding these hazards are provided below.

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

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

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

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

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

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

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

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

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

## 6.0 PROCEDURES

### 6.1 General

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

#### **CAUTION**

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

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

**CAUTION**

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

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

**CAUTION**

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

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

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

## 6.2 Sampling, Monitoring, and Evacuation Equipment

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

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

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

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

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### 6.3 Calculations of Well Volume

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

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

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

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

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

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

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

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## 6.4 Evacuation of Static Water – Purging

### 6.4.1 General

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

### 6.4.2 Evacuation Devices

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

#### Bailers

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

Advantages of bailers include the following:

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

Limitations on the use of bailers include the following:

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

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Safety concerns using a bailer include the following:

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

Control measures for these hazards are provided in Section 6.6.2.

#### Suction Pumps

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

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

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

#### Submersible Pumps

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

Limitations of this class of pumps include the following:

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

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

#### Compressed Gases

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

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

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

#### Electrical Shock

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

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

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To minimize or eliminate electrical generator hazards:

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

#### Lifting Hazards

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

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

#### 6.5 Onsite Water Quality Testing

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

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

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

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

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

### 6.5.1 Measurement of pH

#### 6.5.1.1 General

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

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

#### 6.5.1.2 Principles of Equipment Operation

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

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

#### 6.5.1.3 Equipment

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

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

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

#### 6.5.1.4 Measurement Techniques for Field Determination of pH

##### pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

##### pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

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2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

### **6.5.2 Measurement of Specific Conductance**

#### **6.5.2.1 General**

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

#### **6.5.2.2 Principles of Equipment Operation**

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

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### 6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

### 6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

## 6.5.3 Measurement of Temperature

### 6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

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### 6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

### 6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.
2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.
2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.
3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

## 6.5.4 Measurement of Dissolved Oxygen

### 6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

### 6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

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the two metals, reduction of oxygen to hydroxide ion (OH<sup>-</sup>) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

#### 6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

#### 6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

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7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.
8. Rinse the probe with deionized water.
9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

### **6.5.5 Measurement of Oxidation-Reduction Potential**

#### **6.5.5.1 General**

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

#### **6.5.5.2 Principles of Equipment Operation**

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

#### **6.5.5.3 Equipment**

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential**

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

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2. Thoroughly rinse the electrode with deionized water.
3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

### **6.5.6 Measurement of Salinity**

#### **6.5.6.1 General**

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

#### **6.5.6.2 Principles of Equipment Operation**

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

#### **6.5.6.3 Equipment**

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

#### **6.5.6.4 Measurement Techniques for Salinity**

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the meter before going into the field.

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3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.
5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the probes with deionized water.

### **6.5.7 Measurement of Turbidity**

#### **6.5.7.1 General**

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

#### **6.5.7.2 Principles of Equipment Operation**

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

#### **6.5.7.3 Equipment**

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

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#### 6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

## 6.6 Sampling

### 6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

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Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

#### **6.6.2 Sampling Methods as Related to Low-Flow Sampling**

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:
  - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
  - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
  - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

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during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.
4. Calculate volume of well water to be removed as described in Section 6.3.
5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.
6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.
7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.
8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.
10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.
11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.
12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

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occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.
14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.
15. Process sample containers as described in SOP SA-6.1.
16. Decontaminate equipment as described in SOP SA-7.1.

## **6.7 Low-Flow Purging and Sampling**

### **6.7.1 Scope and Application**

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

### **6.7.2 Equipment**

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

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- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

### 6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).
2. Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.
3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.
4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well
5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

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6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.
  7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
  8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.
  9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.
  10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.
  11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:
    - pH  $\pm 0.2$  standard units
    - Specific conductance  $\pm 10\%$
    - Temperature  $\pm 10\%$
    - Turbidity less than 10 NTUs
    - DO  $\pm 10\%$
  12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.
- NOTE:** VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.
13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

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- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.
- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.
- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.
- Prepare samples for shipping as per SOP SA-6.1.

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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

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**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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**ATTACHMENT A**  
**PURGING EQUIPMENT SELECTION**  
**PAGE 3**

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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 WATER QUALITY TESTING

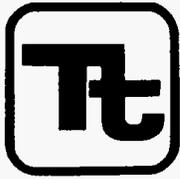
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TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
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

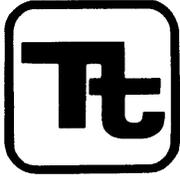
**ORGANIC TESTS:<sup>(8)</sup>**

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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Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T.E. Johnston</i>		

Subject  
FIELD DOCUMENTATION

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

## 2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

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

General personnel qualifications for field documentation activities include the following:

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

## 5.0 PROCEDURES

### 5.1 SITE LOGBOOK

#### 5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

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- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

### **5.1.2 Photographs**

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

## **5.2 FIELD NOTEBOOKS**

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

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separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

### **5.3 FIELD FORMS**

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (<http://intranet.ttnus.com>) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

#### **5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results**

##### **5.3.1.1 Sample Log Sheet**

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

##### **5.3.1.2 Sample Label**

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

##### **5.3.1.3 Chain-of-Custody Record**

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc<sup>®</sup>-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

##### **5.3.1.4 Chain-of-Custody Seal**

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

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and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

#### 5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

### 5.3.2 **Hydrogeological and Geotechnical Forms**

#### 5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

#### 5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

#### 5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

#### 5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

#### 5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

#### 5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

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### 5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

### 5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

### 5.3.3 **Equipment Calibration and Maintenance Form**

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

## 5.4 **FIELD REPORTS**

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

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#### **5.4.1 Daily Activities Report**

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

##### **5.4.1.1 Description**

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

##### **5.4.1.2 Responsibilities**

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

##### **5.4.1.3 Submittal and Approval**

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

#### **5.4.2 Weekly Status Reports**

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

In addition to those described herein, other summary reports may also be contractually required.

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

#### **6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE**

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

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- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

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**ATTACHMENT A  
TYPICAL SITE LOGBOOK ENTRY**

START TIME: \_\_\_\_\_ DATE: \_\_\_\_\_

SITE LEADER: \_\_\_\_\_

PERSONNEL: \_\_\_\_\_

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well \_\_\_\_ resumes. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well \_\_\_\_\_.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well \_\_\_\_\_.
4. Well \_\_\_\_\_ drilled. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 2, page \_\_\_\_ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well \_\_\_\_\_ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manager arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit \_\_\_\_\_.
8. Test pit \_\_\_\_\_ dug with cuttings placed in dump truck. Rig geologist was \_\_\_\_\_. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit \_\_\_\_ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

\_\_\_\_\_  
Field Operations Leader

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**ATTACHMENT B  
SAMPLE LABEL**

	Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		<b>Project:</b>
			<b>Site:</b>
		<b>Location:</b>	
<b>Sample No:</b>		<b>Matrix:</b>	
<b>Date:</b>	<b>Time:</b>	<b>Preserve:</b>	
<b>Analysis:</b>			
<b>Sampled by:</b>		<b>Laboratory:</b>	

Subject

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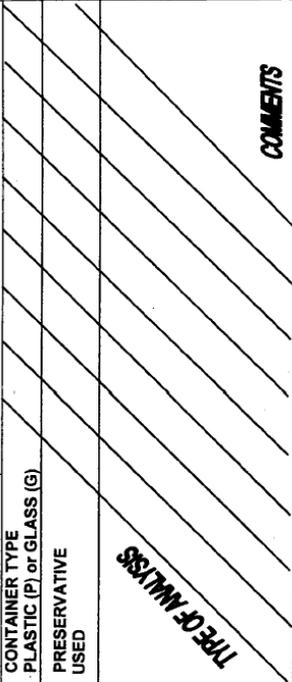
3

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### ATTACHMENT C CHAIN-OF-CUSTODY RECORD FORM

TETRA TECH NUS, INC. CHAIN OF CUSTODY NUMBER 3413 PAGE 1 OF 1

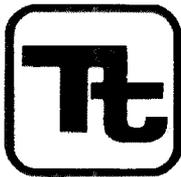
PROJECT NO:		FACILITY:		PROJECT MANAGER		PHONE NUMBER		LABORATORY NAME AND CONTACT:	
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER		PHONE NUMBER		ADDRESS		CITY, STATE	
STANDARD TAT <input type="checkbox"/>		RUSH TAT <input type="checkbox"/>		CARRIER/WAYBILL NUMBER		CONTAINER TYPE		PLASTIC (P) or GLASS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

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY) 4/02R FORM NO. TINUS-001

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**ATTACHMENT 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>
--	--	--



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number DV-04	Page 1 of 8
Effective Date 08/13/01	Revision 0
Applicability Tetra Tech NUS, Inc.	
Prepared Risk Assessment Department	
Approved D. Senovich <i>[Signature]</i>	

Subject  
DATA VALIDATION - NON-CLP INORGANICS FOR  
SOLID AND AQUEOUS MATRICES

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## 1.0 INORGANICS (SW-846 6010B/7470A/7471A/9010A&B/7470/9010)

**Inductively Coupled Plasma Emission Spectroscopy (ICP)** - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

**Graphite Furnace Atomic Absorption Spectroscopy (GFAA)** - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium.

**Cold Vapor Methodology** - Mercury is commonly analyzed using cold vapor methodology.

**Automated Colorimetric Technique** - Cyanide is commonly analyzed using automated colorimetric methodology.

### 1.1 Applicability

These methods are applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

Detection limits for analytes are established on a quarterly basis and are both laboratory and instrument specific.

### 1.2 Data Overview Prior to Validation Process

#### 1.2.1 Data Completeness

The data reviewer must initially verify that all forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required forms will include:

Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Calibration Summary (Form 2A or equivalent).

Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the blank summary (Form 3 or equivalent) (note, filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).

Verify that all ICP analytes are present in both ICSA and ICSAB solutions. Also, verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns.

Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels.

Verify that laboratory duplicate analyses were performed for each matrix. **NOTE:** Field quality control blanks are never to be designated for quality control analyses.

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Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis.

The Method of Standard Additions (MSA) (Form 8 or equivalent) may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 4.1.3.11 for further details.

Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE:** Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12 (or equivalent). Verify that no GFAA analytical results exceed the highest standard in the associated GFAA calibration.

Verify that the Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillation as applicable.

Examine the Form 14s (or equivalent) to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

### **1.3 Technical Evaluation Summary**

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

#### **1.3.1 Holding Times**

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

Metals - 6 months; pH <2  
Mercury - 28 days; pH <2  
Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only. Solid samples do not receive preservative but require maintenance at 4°C (32°C) during shipment and storage.

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The above holding times do not apply to leachate analyses. It is suggested that the data reviewer reference SW-846 Method 1311 for any questions regarding TCLP quality control requirements and analytical procedural requirements; these vary significantly from non-TCLP analyses.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE:** Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

### 1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard
- **GFAA analyses** - must employ a blank and at least three standards. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e.  $r = 0.995$  or greater)
- **Mercury analyses** - must employ a blank and at least three standards ( $r = 0.995$  or greater).
- **Cyanide analyses** - must employ a blank and at least three standards ( $r = 0.995$  or greater). **NOTE:** At least two additional standards (a high or low) must be distilled and compared to similar values on the curve. Values of distilled standards should agree within  $\pm 10\%$  of undistilled standards.

### 1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

The ICV/CCV %R quality control limits are 90-110% for ICP metals, 80-120% for GFAA metals and mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated, (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects remain unaffected. **NOTE:** Qualify results of only those samples associated with the noncompliant ICV or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

### 1.3.4 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours which ever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in a sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration

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present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

### 1.3.5 ICP Interference Check Sample Results

Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. **NOTE:** Affected samples include all samples analyzed between the initial and final solutions or within the eight hour working shift whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 or equivalent and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample, while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\text{Estimated elemental intf.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}] [\text{Conc. Sample}]}{\text{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (**NOTE:** Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

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### 1.3.6 Matrix Spike Sample Analysis (Pre-digestion)

Verify that at least one matrix spike was performed for each matrix for a given set of samples (maximum of 20 samples) within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Any deviations from the referenced method shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant, no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

### 1.3.7 Laboratory Duplicate Precision

Verify that one duplicate sample analysis was performed for each group of samples (maximum of 20 samples) of a similar matrix within an SDG. Control criteria used to evaluate the aqueous laboratory duplicates are as follows:

- a control limit of □20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of □1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of □35% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of □2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

**NOTE:** Review Duplicate Summary (Form 6 or equivalent) carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

### 1.3.8 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances where field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

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- a control limit of  $\leq 30\%$  for relative percent difference when sample and duplicate results are  $>5X$  CRDL
- a control limit of  $\leq 2X$  CRDL for the difference between the sample values when sample and/or duplicate results are  $<5X$  CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a control limit of  $\leq 50\%$  for relative percent difference when sample and duplicate results are  $>5X$  CRDL
- a control limit of  $\leq 4X$  CRDL for the difference between the sample values when sample and/or duplicate results are  $<5X$  CRDL

**NOTE:** The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, field duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

### 1.3.9 Laboratory Control Sample Results

Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R  $<80\%$ , qualify as estimated (J) positive results and (UJ) nondetects, If aqueous LCS %R  $>120\%$ , qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

### 1.3.10 Method of Standard Additions (MSA)

Review MSA Form 8 or equivalent and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r)  $<0.995$ . It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance in the method.

Actions - If calibration correlation coefficient (r)  $<0.995$ , qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.

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### 1.3.11 ICP Serial Dilution Analysis

Verify that all ICP analytes are included on the Form 9 (or equivalent) with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50 IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of suppressed results exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

### 1.3.12 Analysis Run Logs Form 14

The Form 14 or equivalent serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify GFAA PDS percent recoveries within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS % R.

### 1.3.13 Further GFAA Evaluations

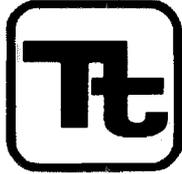
It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

## 1.4 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.



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  
SOIL AND ROCK DRILLING METHODS

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## 1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

## 2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

## 3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

## 4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

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Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

## 5.0 PROCEDURES

### 5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

### 5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

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### 5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.
- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.
- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.
- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotasonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

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also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

### 5.2.2 Continuous-Flight Solid-Stem Auger Drilling

This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous-flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.

There would be very few situations where use of a solid-stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.

### 5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

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and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

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- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

#### **5.2.4 Rotosonic Drilling**

The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.

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The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

### **5.2.5 Reverse Circulation Rotary Drilling**

The common reverse-circulation rig is a water or mud-rotary rig with a large-diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud-rotary). This type of rig is used for the construction of large-capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large-diameter hole which is created. A few special reverse-circulation rotary rigs are made with double-wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.

Advantages of the latter method include:

- The formation water is not contaminated by the drilling water.
- Formation samples can be obtained, from known depths.
- When drilling with air, immediate information is available regarding the water-bearing properties of formations penetrated.
- Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air-rotary rig.

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Disadvantages include:

- Double-wall, reverse-circulation drill rigs are rare and expensive to operate.
- Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.

### 5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

### 5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

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expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

### 5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

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Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

### 5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

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### 5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

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This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

### 5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:
  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."
- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.
- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.
- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.
- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

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- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:
  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.
  - Barge-mounted drill rigs.
  - Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.
  - Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

#### 5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound ( $\pm 2$ -pound) hammer dropping 30 inches ( $\pm 1$  inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

#### 5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.
- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).
- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

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Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotasonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

### 5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

### 5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

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For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.
- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.
- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.
- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

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depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

### **5.5 Materials of Construction**

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

### **5.6 Subsurface Soil Samples**

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

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a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotasonic drilling methods also provide a continuous soil sample.

### **5.7 Rock Sampling (Coring) (ASTM D2113-83)**

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

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**FIGURE 1**

**STANDARD SIZES OF CORE BARRELS AND CASING**

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___ ___/	1 7/8	1	1.875	1.000
BX Wire line ___ ___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___ ___/	3	1 15/16	2.965	1.937

\* All dimensions are in inches; to convert to millimeters, multiply by 25.4.  
 \_\_\_|\_\_\_/ Wire line dimensions and designations may vary according to manufacturer.

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**FIGURE 1**  
**STANDARD SIZES OF CORE BARRELS AND CASING**  
**PAGE TWO**

Size Designations		Casing O.D., Inches	Casing Coupling		Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings		O.D., Inches	I.D., Inches				Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	---	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	---	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	---	5.500			5.650	---	---	---	---
SW	---	6.625			6.790	---	---	---	---
UW	---	7.625			7.800	---	---	---	---
ZW	---	8.625			8.810	---	---	---	---
---	AX ___\	---	---	---	---	1.875	1.750	1.000	---
---	BX ___\	---	---	---	---	2.345	2.250	1.437	---
---	NX ___\	---	---	---	---	2.965	2.813	1.937	---

\* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

\_\_\_\ Wire line dimensions and designations may vary according to manufacturer.

**NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES.**  
**(DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-**  
**D-2889**

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### 5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.
- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.
- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.
- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.
- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

### 5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

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After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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**ATTACHMENT A**  
**DRILLING EQUIPMENT SIZES**

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	---
	7 1/4	6 1/4	3 1/4	---
	13 1/4	12	6	---
Thin Wall Tube Samplers (Ref. 7)	---	2	1 7/8	---
	---	2 1/2	2 3/8	---
	---	3	2 7/8	---
	---	3 1/2	3 3/8	---
	---	4 1/2	4 3/8	---
	---	5	4 3/4	---
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

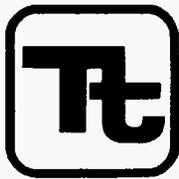
\* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

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**ATTACHMENT A  
DRILLING EQUIPMENT SIZES  
PAGE TWO**

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

\*\* Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

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Applicability	Tetra Tech NUS, Inc.		
Prepared	Health & Safety		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
UTILITY LOCATING AND EXCAVATION CLEARANCE

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## 1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

## 2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

## 3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

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#### 4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

#### 5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

##### 5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.
- 2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scars and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

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locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.
4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.
5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain
6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.
7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.
8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

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**5.2 Overhead Power Lines**

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

<u>Nominal Voltage</u>	<u>Minimum Clearance</u>
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

**6.0 UNDERGROUND LOCATING TECHNIQUES**

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

**6.1 Geophysical Methods**

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

**Electromagnetic Induction**

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

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## **Magnetics**

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

## **Ground Penetrating Radar**

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

### **6.2 Passive Detection Surveys**

#### **Acoustic Surveys**

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

#### **Thermal Imaging**

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

### **6.3 Intrusive Detection Surveys**

#### **Vacuum Excavation**

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

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debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

### Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

### Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

## 7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.
2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.
4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

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5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

## 8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4  
 OSHA 29 CFR 1926(b)(2)  
 OSHA 29 CFR 1926(b)(3)  
 TtNUS Utility Locating and Clearance Policy  
 TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction  
 TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys  
 TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

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**ATTACHMENT 1  
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES**



**American Public Works Association**  
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625  
Phone (816) 472-6100 • Fax (816) 472-1610  
Web www.apwa.net • E-mail apwa@apwa.net

**ONE-CALL SYSTEMS INTERNATIONAL  
CONDENSED DIRECTORY**

- |  |   |  |
|--|---|--|
| <p><b>Alabama</b><br/>Alabama One-Call<br/>1-800-292-8525</p> <p><b>Alaska</b><br/>Locate Call Center of Alaska, Inc.<br/>1-800-478-3121</p> <p><b>Arizona</b><br/>Arizona Blue Stake<br/>1-800-782-5348</p> <p><b>Arkansas</b><br/>Arkansas One Call System, Inc.<br/>1-800-482-8998</p> <p><b>California</b><br/>Underground Service Alert North<br/>1-800-227-2600<br/>Underground Service Alert of Southern<br/>California<br/>1-800-227-2600</p> <p><b>Colorado</b><br/>Utility Notification Center of Colorado<br/>1-800-922-1987</p> <p><b>Connecticut</b><br/>Call Before You Dig<br/>1-800-922-4455</p> <p><b>Delaware</b><br/>Miss Utility of Delmarva<br/>1-800-282-8555</p> <p><b>Florida</b><br/>Sunshine State One-Call of Florida, Inc.<br/>1-800-432-4770</p> <p><b>Georgia</b><br/>Underground Protection Center, Inc.<br/>1-800-282-7411</p> <p><b>Hawaii</b><br/>Underground Service Alert North<br/>1-800-227-2600</p> <p><b>Idaho</b><br/>Dig Line Inc.<br/>1-800-342-1585<br/>Kootenai County One-Call<br/>1-800-428-4950<br/>Shoshone - Benewah One-Call<br/>1-800-398-3285</p> <p><b>Illinois</b><br/>JULIE, Inc.<br/>1-800-892-0123<br/>Digger (Chicago Utility Alert Network)<br/>312-744-7000</p> <p><b>Indiana</b><br/>Indiana Underground Plant Protection<br/>Service<br/>1-800-382-5544</p> | <p><b>Iowa</b><br/>Iowa One-Call<br/>1-800-292-8989</p> <p><b>Kansas</b><br/>Kansas One-Call System, Inc.<br/>1-800-344-7233</p> <p><b>Kentucky</b><br/>Kentucky Underground Protection Inc.<br/>1-800-752-6007</p> <p><b>Louisiana</b><br/>Louisiana One Call System, Inc.<br/>1-800-272-3020</p> <p><b>Maine</b><br/>Dig Safe System, Inc.<br/>1-888-344-7233</p> <p><b>Maryland</b><br/>Miss Utility<br/>1-800-257-7777<br/>Miss Utility of Delmarva<br/>1-800-282-8555</p> <p><b>Massachusetts</b><br/>Dig Safe System, Inc.<br/>1-888-344-7233</p> <p><b>Michigan</b><br/>Miss Dig System, Inc.<br/>1-800-482-7171</p> <p><b>Minnesota</b><br/>Gopher State One Call<br/>1-800-252-1168</p> <p><b>Mississippi</b><br/>Mississippi One-Call System, Inc.<br/>1-800-227-6477</p> <p><b>Missouri</b><br/>Missouri One-Call System, Inc.<br/>1-800-344-7483</p> <p><b>Montana</b><br/>Utilities Underground Protection Center<br/>1-800-424-5555<br/>Montana One Call Center<br/>1-800-551-8344</p> <p><b>Nebraska</b><br/>Diggers Hotline of Nebraska<br/>1-800-331-5666</p> <p><b>Nevada</b><br/>Underground Service Alert North<br/>1-800-227-2600</p> <p><b>New Hampshire</b><br/>Dig Safe System, Inc.<br/>1-888-344-7233</p> | <p><b>New Jersey</b><br/>New Jersey One Call<br/>1-800-272-1000</p> <p><b>New Mexico</b><br/>New Mexico One Call System, Inc.<br/>1-800-321-2537<br/>Las Cruces- Dona Ana Blue Stakes<br/>1-888-526-0400</p> <p><b>New York</b><br/>Dig Safely New York<br/>1-800-862-7962<br/>New York City- Long Island One Call<br/>Center<br/>1-800-272-4480</p> <p><b>North Carolina</b><br/>The North Carolina One-Call Center,<br/>Inc.<br/>1-800-632-4949</p> <p><b>North Dakota</b><br/>North Dakota One-Call<br/>1-800-795-0555</p> <p><b>Ohio</b><br/>Ohio Utilities Protection Service<br/>1-800-362-2764<br/>Oil &amp; Gas Producers Underground<br/>Protect'n Svc<br/>1-800-925-0988</p> <p><b>Oklahoma</b><br/>Call Okie<br/>1-800-522-6543</p> <p><b>Oregon</b><br/>Oregon Utility Notification Center/One<br/>Call Concepts<br/>1-800-332-2344</p> <p><b>Pennsylvania</b><br/>Pennsylvania One Call System, Inc.<br/>1-800-242-1776</p> <p><b>Rhode Island</b><br/>Dig Safe System, Inc.<br/>1-888-344-7233</p> <p><b>South Carolina</b><br/>Palmetto Utility Protection Service Inc.<br/>1-888-721-7877</p> <p><b>South Dakota</b><br/>South Dakota One Call<br/>1-800-781-7474</p> <p><b>Tennessee</b><br/>Tennessee One-Call System, Inc.<br/>1-800-351-1111</p> |
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**ATTACHMENT 1 (Continued)**

**Texas**

Texas One Call System  
1-800-245-4545  
Texas Excavation Safety System, Inc.  
1-800-344-8377  
Lone Star Notification Center  
1-800-669-8344

**Utah**

Blue Stakes of Utah  
1-800-662-4111

**Vermont**

Dig Safe System, Inc.  
1-888-344-7233

**Virginia**

Miss Utility of Virginia  
1-800-552-7001  
Miss Utility (Northern Virginia)  
1-800-257-7777

**Washington**

Utilities Underground Location Center  
1-800-424-5555  
Northwest Utility Notification Center  
1-800-553-4344  
Inland Empire Utility Coordinating  
Council  
509-456-8000

**West Virginia**

Miss Utility of West Virginia, Inc.  
1-800-245-4848

**Wisconsin**

Diggers Hotline, Inc.  
1-800-242-8511

**Wyoming**

Wyoming One-Call System, Inc.  
1-800-348-1030  
Call Before You Dig of Wyoming  
1-800-849-2476

**District of Columbia**

Miss Utility  
1-800-257-7777

**Alberta**

Alberta One-Call Corporation  
1-800-242-3447

**British Columbia**

BC One Call  
1-800-474-6886

**Ontario**

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

**Quebec**

Info-Excavation  
1-800-663-9228

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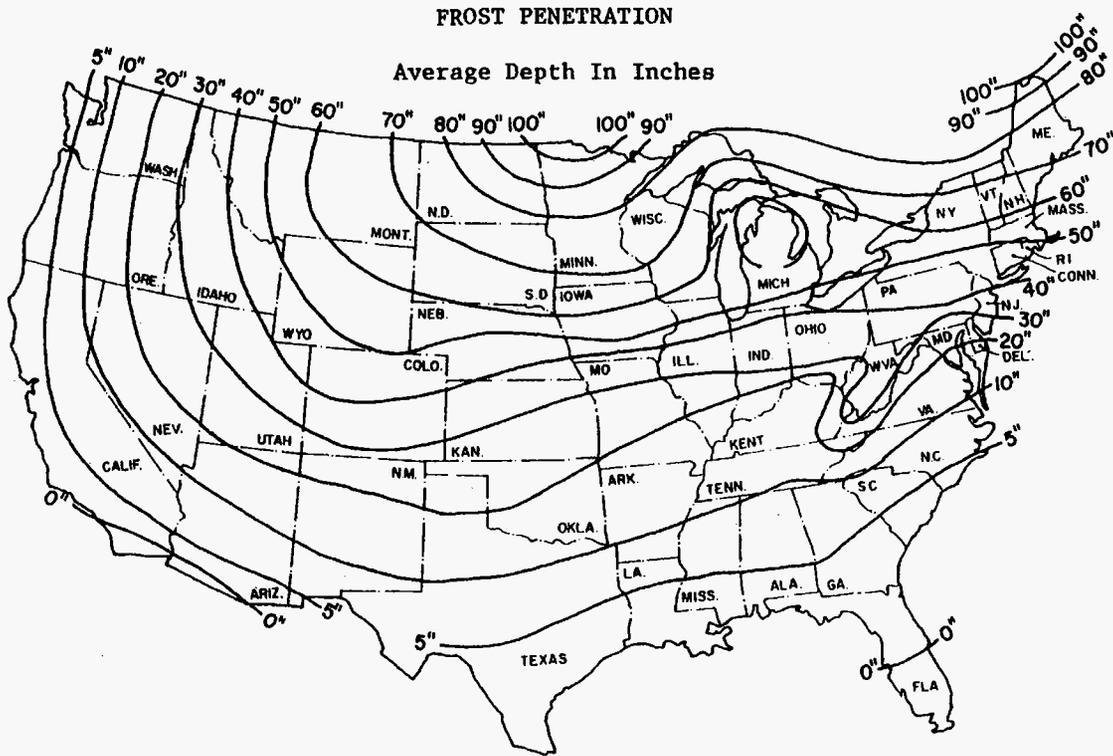
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### ATTACHMENT 2

### FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

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**ATTACHMENT 3  
UTILITY CLEARANCE FORM**

Client: \_\_\_\_\_ Project Name: \_\_\_\_\_  
Project No.: \_\_\_\_\_ Completed By: \_\_\_\_\_  
Location Name: \_\_\_\_\_ Work Date: \_\_\_\_\_  
Excavation Method/Overhead Equipment: \_\_\_\_\_

1. Underground Utilities Circle One
- a) Review of existing maps? yes no N/A
  - b) Interview local personnel? yes no N/A
  - c) Site visit and inspection? yes no N/A
  - d) Excavation areas marked in the field? yes no N/A
  - e) Utilities located in the field? yes no N/A
  - f) Located utilities marked/added to site maps? yes no N/A
  - g) Client contact notified yes no N/A  
Name \_\_\_\_\_ Telephone: \_\_\_\_\_ Date: \_\_\_\_\_
  - g) State One-Call agency called? yes no N/A  
Caller: \_\_\_\_\_  
Ticket Number: \_\_\_\_\_ Date: \_\_\_\_\_
  - h) Geophysical survey performed? yes no N/A  
Survey performed by: \_\_\_\_\_  
Method: \_\_\_\_\_ Date: \_\_\_\_\_
  - i) Hand excavation performed (with concurrent use of utility  
detection device)? yes no N/A  
Completed by: \_\_\_\_\_  
Total depth: \_\_\_\_\_ feet Date: \_\_\_\_\_
  - j) Trench/excavation probed? yes no N/A  
Probing completed by: \_\_\_\_\_  
Depth/frequency: \_\_\_\_\_ Date: \_\_\_\_\_

2. Overhead Utilities Present Absent
- a) Determination of nominal voltage yes no N/A
  - b) Marked on site maps yes no N/A
  - c) Necessary to lockout/insulate/re-route yes no N/A
  - d) Document procedures used to lockout/insulate/re-route yes no N/A
  - e) Minimum acceptable clearance (SOP Section 5.2): \_\_\_\_\_

3. Notes:  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Approval:  
\_\_\_\_\_  
Site Manager/Field Operations Leader Date

c: PM/Project File  
Program File

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**ATTACHMENT 4  
OSHA LETTER OF INTERPRETATION**

Mr. Joseph Caldwell  
Consultant  
Governmental Liaison  
Pipeline Safety Regulations  
211 Wilson Boulevard  
Suite 700  
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

***Question:** Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

*Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?*

**Answer**

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651 (Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours \* \* \* or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

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#### ATTACHMENT 4 (Continued)

When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, “acceptable means” must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either “other acceptable means” or “safe and acceptable means.” The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified “careful probing or hand digging” as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language “to allow other, *equally effective means* of locating such installations.” The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used where the utilities have not been marked and detection equipment is not being used – “probing with hand-held tools.” This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

OSHA received two comments \* \* \* and input from ACCSH [OSHA’s Advisory Committee on Construction Safety and Health] \* \* \* on this provision. All commenters recommended dropping ‘such as probing with hand-held tools’ from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of “acceptable means” in the final provision.

#### Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a “shooter” (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an “acceptable means” for locating underground utilities.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 15 of 15
	Revision 2	Effective Date 12/03

**ATTACHMENT 4 (Continued)**

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director  
Directorate of Construction

**NOTE:** OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA's interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at <http://www.osha.gov>.

**APPENDIX B**

**FIELD FORMS**

# 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  <hr/> <hr/>

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



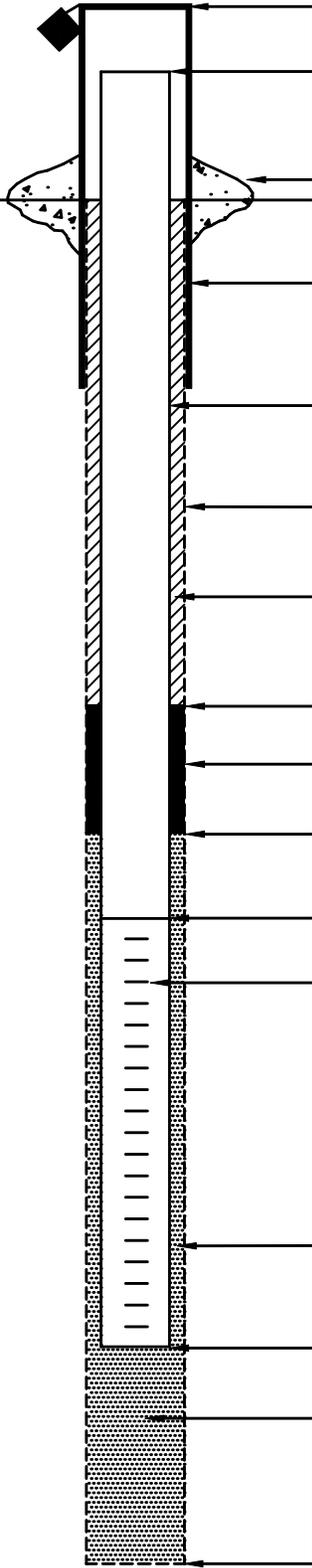
Tetra Tech NUS, Inc.

# OVERBURDEN MONITORING WELL SHEET STICK-UP

WELL NO.: \_\_\_\_\_

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

ACAD:FORM\_MWSU.dwg 07/20/99 INL



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: \_\_\_\_\_ / \_\_\_\_\_

ELEVATION/HEIGHT OF TOP OF RISER PIPE: \_\_\_\_\_ / \_\_\_\_\_

TYPE OF SURFACE SEAL: \_\_\_\_\_

I.D. OF SURFACE CASING: \_\_\_\_\_

TYPE OF SURFACE CASING: \_\_\_\_\_

RISER PIPE I.D.: \_\_\_\_\_

TYPE OF RISER PIPE: \_\_\_\_\_

BOREHOLE DIAMETER: \_\_\_\_\_

TYPE OF BACKFILL: \_\_\_\_\_

ELEVATION/DEPTH TOP OF SEAL: \_\_\_\_\_ / \_\_\_\_\_

TYPE OF SEAL: \_\_\_\_\_

DEPTH TOP OF SAND PACK: \_\_\_\_\_

ELEVATION/DEPTH TOP OF SCREEN: \_\_\_\_\_ / \_\_\_\_\_

TYPE OF SCREEN: \_\_\_\_\_

SLOT SIZE x LENGTH: \_\_\_\_\_

I.D. OF SCREEN: \_\_\_\_\_

TYPE OF SAND PACK: \_\_\_\_\_

ELEVATION/DEPTH BOTTOM OF SCREEN: \_\_\_\_\_ / \_\_\_\_\_

ELEVATION/DEPTH BOTTOM OF SAND PACK: \_\_\_\_\_ / \_\_\_\_\_

BACKFILL MATERIAL BELOW SAND: \_\_\_\_\_

ELEVATION/DEPTH OF HOLE: \_\_\_\_\_ / \_\_\_\_\_



Tetra Tech NUS, Inc.

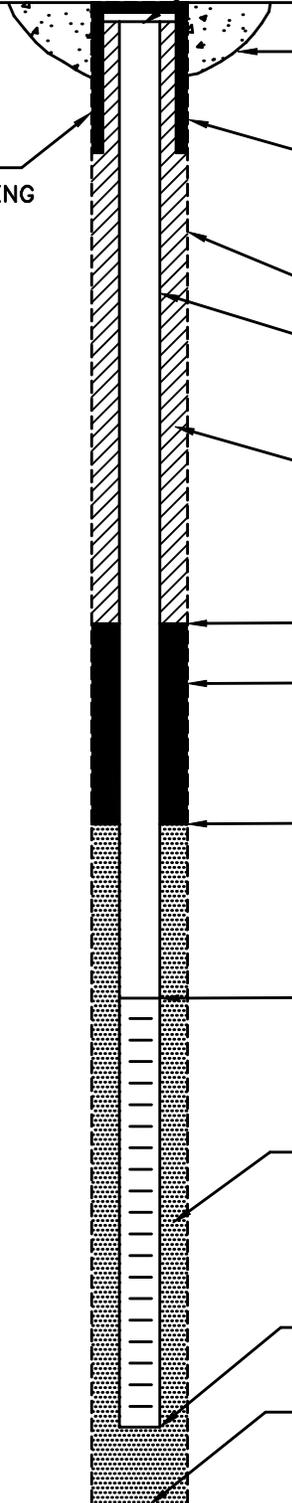
# OVERBURDEN MONITORING WELL SHEET FLUSH - MOUNT

WELL NO.: \_\_\_\_\_

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

ACAD:FORM\_MWFM.dwg 07/20/99 INL

FLUSH MOUNT  
SURFACE CASING  
WITH LOCK



ELEVATION TOP OF RISER: \_\_\_\_\_

TYPE OF SURFACE SEAL: \_\_\_\_\_

TYPE OF PROTECTIVE CASING: \_\_\_\_\_

I.D. OF PROTECTIVE CASING: \_\_\_\_\_

DIAMETER OF HOLE: \_\_\_\_\_

TYPE OF RISER PIPE: \_\_\_\_\_

RISER PIPE I.D.: \_\_\_\_\_

TYPE OF BACKFILL/SEAL: \_\_\_\_\_

ELEVATION/DEPTH TOP OF SEAL: \_\_\_\_\_ / \_\_\_\_\_

TYPE OF SEAL: \_\_\_\_\_

ELEVATION/DEPTH TOP OF SAND: \_\_\_\_\_ / \_\_\_\_\_

ELEVATION/DEPTH TOP OF SCREEN: \_\_\_\_\_ / \_\_\_\_\_

TYPE OF SCREEN: \_\_\_\_\_

SLOT SIZE x LENGTH: \_\_\_\_\_

TYPE OF SAND PACK: \_\_\_\_\_

DIAMETER OF HOLE IN BEDROCK: \_\_\_\_\_

ELEVATION / DEPTH BOTTOM OF SCREEN: \_\_\_\_\_ / \_\_\_\_\_

ELEVATION / DEPTH BOTTOM OF SAND: \_\_\_\_\_ / \_\_\_\_\_

ELEVATION/DEPTH BOTTOM OF HOLE: \_\_\_\_\_ / \_\_\_\_\_

BACKFILL MATERIAL BELOW SAND: \_\_\_\_\_



# CONFINING LAYER MONITORING WELL SHEET

WELL NO.: \_\_\_\_\_

**Tetra Tech NUS, Inc.**

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

ACAD:FORM\_CLMW.dwg 07/20/99 INL

The diagram shows a vertical cross-section of a monitoring well. From top to bottom, it features a surface casing with a seal, a riser pipe, a permeable casing, a seal, a sand pack, and a screen. A cross-hatched 'CONFINING LAYER' is shown between the permeable casing and the seal. The borehole diameter is shown to be larger than the casing diameter below the casing. A north arrow is located at the top left of the well diagram.

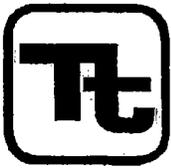
ELEVATION/HEIGHT OF TOP OF SURFACE CASING:	_____ / _____
ELEVATION/HEIGHT OF TOP OF RISER PIPE:	_____ / _____
ELEVATION/HEIGHT OF TOP OF PERM. CASING:	_____ / _____
TYPE OF SURFACE SEAL:	_____
I.D. OF SURFACE CASING:	_____
TYPE OF SURFACE CASING:	_____
RISER PIPE I.D.:	_____
TYPE OF RISER PIPE:	_____
BOREHOLE DIAMETER:	_____
PERM. CASING I.D.:	_____
TYPE OF CASING AND BACKFILL:	_____
ELEVATION/DEPTH TOP CONFINING LAYER:	_____ / _____
ELEVATION/DEPTH BOTTOM OF CASING:	_____ / _____
ELEVATION/DEPTH BOTTOM CONFINING LAYER:	_____ / _____
ELEVATION/DEPTH TOP OF SEAL:	_____ / _____
TYPE OF SEAL:	_____
DEPTH TOP OF SAND PACK:	_____
ELEVATION/DEPTH TOP OF SCREEN:	_____ / _____
TYPE OF SCREEN:	_____
TYPE OF SAND PACK:	_____
BOREHOLE DIA. BELOW CASING:	_____
ELEVATION/DEPTH BOTTOM OF SCREEN:	_____ / _____
ELEVATION/DEPTH BOTTOM OF SAND PACK:	_____ / _____
BACKFILL MATERIAL BELOW SAND:	_____
ELEVATION/DEPTH OF HOLE:	_____ / _____











**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.



# 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 :

	Layer 1	Layer 2	Layer 3
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:
---	-----------

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_





**APPENDIX C**

**LABORATORY  
STANDARD OPERATING PROCEDURES**

## 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**

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**19**

**APPROVED BY:**

**SECTION MANAGER**

**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 (HNO<sub>3</sub>). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). 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 CLP-IIM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO<sub>3</sub>, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO<sub>3</sub> and 5% HCl. Use 0.125 mLs HNO<sub>3</sub> and 0.3125 mLs HCl to each 50 mL vessel.

#### 2. Solids

a. A 1.0  $\pm$ 0.02 gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and recorded in the sample digestion logbook.

### 3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

### B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

## VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 85° for oil samples. Record in digestion logbook.

## IX. PROCEDURE

### A. Glassware preparation for oil digestion or when the hot-block can not be used:

1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
2. Rinse glassware with reagent water that contains 5% HNO<sub>3</sub> and 5% HCl followed by a rinse with reagent water.
3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

### B. Aqueous sample filtration (for dissolved metals):

1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO<sub>3</sub> followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
2. Rinse a 0.45 micron filter with 1:5 HNO<sub>3</sub> thoroughly, followed by D.I. water.
3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
4. Discard the first 50 to 100 mL.
5. A preparation blank must be taken through the filtration step and analyzed with the sample.
6. Preserve the sample with HNO<sub>3</sub> to pH<2.
7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO<sub>3</sub>. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

### C. Aqueous sample preparation

1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
- b. Add 0.50 mL ( 1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO<sub>3</sub> to the sample.
- c. Add 2.5 mL ( 5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
- d. Cover the sample with a ribbed watch glass or equivalent source.
- e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, ( 12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
- f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- g. Bring sample to its predigestion volume ( or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.
- h. The sample is now ready for analysis.
- i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

## 2 Method 200.7, "Acid digestion procedure for total recoverable metals".

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
  - b. Add 1.0 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Add 2.50 mL concentrated HCl to the sample.
  - d. Cover the sample with a ribbed watch glass or equivalent source.
  - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
  - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
  - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
  - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
  - i. The sample is now ready for analysis.
  - j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel.
  - b. Add 1.5 mL concentrated HNO<sub>3</sub> to the sample.
  - c. Cover the sample with a ribbed watch glass.
  - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the log book. Take the volume down to a low

volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.

- e. Add another 1.5 mL portion of concentrated HNO<sub>3</sub> to the sample.
- f. Cover the sample with a ribbed watch glass.
- g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
- h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
- i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
- j. Cover the digestion vessel and reflux for an additional 15 minutes.
- k. Bring sample to its predigestion volume in digestion vessel.
- l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

**Note:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

- m. The sample is now ready for analysis.
  - n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"

- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
- b. Add 2.5 mL 1:1 HCl to the sample.
- c. Heat 15 minutes in a hot bath.
- d. Filter through a membrane filter.
- e. Adjust filtrate volume to 50 mL with DI water.
- f. Transfer to ICP analyst.

#### D. Solid sample preparation

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

#### **Grinding of Vegetation Samples**

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill

or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"

- a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
- b. Add 10 mL of 1:1 nitric acid ( $\text{HNO}_3$ ), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated  $\text{HNO}_3$ , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
- c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
- d. Continue to add 30%  $\text{H}_2\text{O}_2$  in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30%  $\text{H}_2\text{O}_2$ .)
- e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated  $\text{HNO}_3$ , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v)  $\text{HNO}_3$ . The sample is now ready for analysis.

- f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, “**Acid digestion of Sediments, Sludges and Soils**”

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

**NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.**

- c. Add 5 mL D.I. water and 5 mL concentrated  $\text{HNO}_3(1:1)$ , mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at  $95^\circ\text{C}$  for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book
- d. Allow the sample to cool. Add 5 mL concentrated  $\text{HNO}_3$ , replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by  $\text{HNO}_3$ , repeat this step (addition of 5 mL of concentrated  $\text{HNO}_3$ ) over and over until no brown fumes are given off by the sample indicating the complete reaction with  $\text{HNO}_3$ . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at  $95^\circ\text{C} \pm 5^\circ\text{C}$  for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of  $\text{H}_2\text{O}_2$  if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H<sub>2</sub>O<sub>2</sub> to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H<sub>2</sub>O<sub>2</sub>.)

- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
- g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
- h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

**NOTE1:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

**NOTE2:** To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

- j. The sample is now ready for analysis.
- k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## E. Oils

## 1. Method 3031, "Digestion Procedure for Oils"

**NOTE: THIS METHOD IS VERY TIME CONSUMING--  
DISCUSS SUB-CONTRACTING SAMPLES WITH  
YOUR SUPERVISOR AS SOON AS THEY COME IN  
THE DOOR.**

- a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.
  
- g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to potassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

- h. Cautiously add 1.0 mL concentrated H<sub>2</sub>SO<sub>4</sub>, and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The H<sub>2</sub>SO<sub>4</sub> can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

NOTE: To prevent a strong exothermic reaction, H<sub>2</sub>SO<sub>4</sub> 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 any ammonium chloride formed will solidify. If this occurs, either add enough water to dissolve the solids or filter out the solids and wash the residue with deionized water. The filtrate can be analyzed by ICP-AES.
- u. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## **X. CALCULATIONS**

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

## **XI. QUALITY CONTROL**

- A. Digestion

1. Temperature blank
  - a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
  - b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
  
2. Blanks
  - a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples. **NOTE: The blank for OILs MUST include an analyte-free oil or explosive reactions can occur.**
  - b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
  - c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
  - d. Sample is given a unique identifier in the digestion log.
  
3. Laboratory Control Samples
  - a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
  - b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
  - c. For soil/sediment samples, a soil matrix standard reference material (SRM ) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
  - d. Sample is given a unique identifier in the digestion log.
  - e. Recoveries of standard reference materials or laboratory control samples spiked with organo-metallic standards recoveries should be **±25% of their true values for OILS.**

#### 4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

**NOTE:** Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

#### 5. Blank Spike

- a. This is required for certain projects.

### B. Sample Matrix

**NOTE:** Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

#### 1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

**NOTE:** For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium  
Magnesium  
Sodium  
Potassium

- v. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)

**d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

## XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
  - 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
  - 1. Redigest another sample aliquot.
  - 2. Glass beaker dry for an extended period of time? Discard beaker.

## XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
  - 1. All metals glassware must be kept separate from all other laboratory glassware.
  - 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
  - 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
  
- F. Please consult Waste Disposal SOP-405, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### **Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT**

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

**DEFINITIONS** – Refer to SOP-431 for common environmental laboratory definitions.

**MERCURY ANALYSIS IN WATER**  

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**BY MANUAL COLD VAPOR TECHNIQUE**  

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**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**  

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**SOP NUMBER:** **SOP-103**  

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**REVISION NUMBER:** **16**  

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**APPROVED BY:**  

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**SECTION MANAGER**

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**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** **01/28/09**  

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**DATE OF LAST REVIEW** **01/28/09**  

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## MERCURY ANALYSIS IN WATER BY MANUAL COLD VAPOR

### References:

**SW846 Method 7470A**  
**USEPA Method 245.1**  
**USEPA SOW ILM04.1**  
**See Addendum for SOW ILM05.2**

## I. SCOPE AND APPLICATION

- A. This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.
- B. In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.
- C. The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

## II. SUMMARY OF METHOD

- A. The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

### III. SAMPLE HANDLING AND PRESERVATION

- A. Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.
- B. The holding time for the mercury digestion is 28 days from time of sampling.

### IV. INTERFERENCES

- A. Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.
- B. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.
- C. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).
- D. Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

### V. Safety

- A. Normal accepted laboratory practices should be followed while performing this procedure.
- B. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

- C. Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.
- D. The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

## VI. EQUIPMENT/APPARATUS

- A. Perken Elmer Flow injection Mercury system
- B. Mod Block Digester set to maintain  $95\pm 2^{\circ}\text{C}$  for 2 hours.
- C. Polypropylene sample digestion vessels with snap or screw caps or equivalent.  
**Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.**

## VII. REAGENTS AND STANDARD PREPARATION

### A. REAGENTS

1. Concentrated sulfuric acid suitable for Hg determination.
2. Concentrated nitric acid suitable for Hg determination.
3. Stannous chloride: in a 1000 mL volumetric flask add approximately 500 mL D.I. water, 30 mL concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
4. 3% HCl Carrier Solution: Dilute 30 mL of concentrated metals grade HCl to one liter. Prepare fresh daily.
5. Sodium chloride-hydroxylamine chloride solution: dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.
5. Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mL of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring ( minimum of 3 hours if absolutely necessary ). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.

6. Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mL D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

## B. STANDARDS

### 1. Traceability

- a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
- c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

**NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes ( or calibrated Eppendorfs ). All standards, blanks, and samples are taken through the digestion process.

- a. Stock mercury solution: (100  $\mu\text{g/mL}$ ). Order from manufacturer already prepared. This solution is given a unique identifier.
- b. Primary source and secondary source mercury standard solutions at 200  $\mu\text{g/L}$ : dilute 2 mL of stock solution to 1000 mL in a 1000 mL volumetric flask, with 1.5 mL concentrated  $\text{HNO}_3$ .
- c. Calibration standards
  - i. Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

- a. Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

<u>ug/L Hg</u>	<u>mLs of 200 ug/L standard in 30 mLs</u>
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

- iii. Appropriate reagents are added as below in the sample preparation section.
- iv. Prepare one vessel for each.
- v. It is necessary to digest the calibration standards.
- e. Calibration verification standards
- i. Initial calibration verification ( ICV ) solution – 4.0 ug/L
- a. Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)
- b. Appropriate reagents are added as below in the sample preparation section.
- c. It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.
- ii. Continuing calibration verification ( CCV ) solution
- a. Prepared from the primary source standard.
- b. Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

- c. Appropriate reagents are added as below in the sample preparation section.
  - d. It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.
- f. Digestion standards
- i. Laboratory control sample
    - a. Prepared from the secondary source standard.
    - b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.
    - c. Appropriate reagents are added as below in the sample preparation section.
    - d. This solution should be given a unique identifier in the digestion log.
  - ii. Matrix Spikes
    - a. Prepared from the secondary source working standard.
    - b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.
    - c. Appropriate reagents are added as below in the sample preparation section.

## VIII. CALIBRATION

A. Set up the instrument with proper operating parameters.

1. Perkin Elmer Flow Injection Mercury System (FIMS).

- i. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. (**Perkin-Elmer tygon**

**tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)**

- ii. Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)
- iii. Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)
- iv. Turn on computer and go to icon "AA Win LAB Analyst".
- v. Go to method; select "Hg CAL 2" then OK.
- vi. Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.
- vii. Go to "Sample Info" and enter the order of the samples and other information that may be needed.
- viii. Save entered sample list under "Save ....sample info file" Note: description and batch ID are normally the date of analysis.
- ix. Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.
- x. Turn the printer on.
- xi. Connect all tubing to the pump and blocks.
- xii. Start the pump by going to "FIAS" and click the pump 1 Icon (120).
- xiii. The pump will start, then lock down and tighten the tubes onto the pump.
- xiv. Turn on the nitrogen tank, it should be above 500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.
- xv. The pressure gauge on the PE100 should be just below 100.
- xvi. Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

- xvii. Adjust the spring tension tubing until there is a constant “bubble of low rate” coming out to the waste tube.
- xviii. Place carrier tubes into carrier and stannous chloride tube into SnCl<sub>2</sub>. (click valve fill inject and make sure flow is correct and the line is rinsed).
- xix. Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.
- xx. Allow a few minutes for reagents to flow through the system before starting analysis.
- xxi. Calibrate: Go to “Auto” click on “Analyze”, click on “calibrate”.
- xxii. “Select Location” enter #'s to be ran, and then press “OK”. Samples are done in increments of 10 samples

B. Analyze the calibration standards as below.

- 1. New calibration points must be analyzed when the ICV analysis is not within  $\pm 5\%$ . **A curve must be analyzed daily for all projects especially USACE and CLP projects.**
- 2. The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of  $\geq 0.995$  ( USACE ) or 0.998 ( other ). If not, a new curve must be analyzed.

## IX. PROCEDURE

A. Glassware preparation

- 1. After use, samples are neutralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.
- 2. Acid clean the glassware used for mercury prep as follows:
  - a. Rinse with low Hg content 1:1 HCl.
  - b. Rinse with D.I. water.

B. Label the vessels indicating which sample will be in each..

C. Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the digestion log.

D. Sample preparation

1. Transfer 30 mL, or an aliquot diluted to 30 mL of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

**NOTE:** Normally, an automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix get spiked. Check with your manager.

2. Add 1.5 mL of concentrated sulfuric acid to each vessel and mix.
3. Add 0.75 mL of concentrated nitric acid to each bottle and mix.
4. Add 4.5 mL potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mL). If the purple color does not persist after the addition of 7.5 mL  $\text{KMnO}_4$  the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

**NOTE:** The same amount of  $\text{KMnO}_4$  added to the samples should be present in the standards and blanks.

5. Add 2.4 mL of potassium persulfate to each vessel and mix. Cover.
6. Heat for 2 hours in the block digester at  $95 \pm 2^\circ\text{C}$  ( the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature ), cool.
6. Samples may be saved at this point if there is not time to run the whole set that day.

**NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.**

E. Sample analysis

1. Set up the instrument as described in the calibration section above.
2. When ready to run samples, add 1.8 mL of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of  $\pm 10\%$  for SW846-7470 and  $\pm 5\%$  for 245.1. Followed by the ICB ( $< \pm MDL$  for USACE or  $\pm RL/CRDL$  for others and CLP).
3. Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of  $\pm 20\%$  for SW846-7470 and  $\pm 10\%$  for 245.1
4. CCB must always follow the CCV. Control limits are ( $< \pm MDL$  for USACE or  $\pm RL/CRDL$  for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected  $> 2xMDL$  for DOD QSM Ver. 3.**
5. The autosampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	ICV
10	ICB
11	LCSW
AS LOC	Sample ID
12	PBW
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	CCV

22	CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	FCV
34	FCB

F. Data reporting

1. Reduce data to result which will be reported.
2. Complete the data review checklist ( attached ). Must be completed and attached to each set of USACE data.

**X. CALCULATIONS**

- A. Apply a least squares fit to the calibration standards plotting  $\mu\text{g Hg/L}$  versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume ( the 0.1  $\mu\text{g Hg}$  standard will be input as 1.0  $\mu\text{g Hg/L}$  ) ( 0.1 $\mu\text{g Hg}$  / 0.030 L solution ).
- B. Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.
- C. Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.
- D. Report the data as  $\mu\text{g Hg/L}$  of sample.

**XI. QUALITY CONTROL (Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)**

A. Daily

1. **The instrument must be calibrated daily for all projects.**

2. Begin each analysis with an ICV(QCS) second source. The control limits are  $\pm 10\%$  and IPC(CCV) for 245.1, limits are  $\pm 5\%$  and subsequent analyses are  $\pm 10\%$ .
  3. Analyze ICB. Control limits ( $<\pm$ MDL for USACE or  $\pm$ RL/CRDL for others and CLP)., depending on method. **No analyte detected  $>2x$ MDL for DOD QSM Ver. 3.**
  4. If the ICV(QCS) is not in control a new curve must be analyzed prior to sample analysis.
  5. If the IPC(initial CCV) for 245.1 is not within the limits of  $\pm 5\%$ , try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.
  6. Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are  $\pm 20\%$  for SW846-7470 and  $\pm 10\%$  for 245.1.
  7. A CCB must always follow a CCV, the control limit is ( $<\pm$ 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  $>2x$ MDL for DOD QSM Ver. 3.**
- B. Quarterly or as needed when doing straight CLP work.
1. IDL's for CLP 4.1.
- C. Digestion
1. LCS data should be maintained and available for easy reference or inspection.
  2. Preparation blank ( $<1/2$   $\pm$ RL or  $\pm$ RL/CRDL for common contaminants (DOD) and  $\pm$ RL/CRDL for others and CLP).
    - a. Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the

preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.

3. Laboratory control sample ( LCS )
  - a. Employ a minimum of one laboratory control sample ( LCS ) per sample batch to verify the digestion procedure. The LCS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are  $\pm 20\%$  for SW846-7470 and  $\pm 15\%$  for 245.1. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified. The project manager or QA Officer will make this determination.

#### D. Sample matrix

1. Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. CLP does not allow this. Project specific requirements will take precedence in these situations.
2. Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within  $\pm 25\%$  of the true value ( **$\pm 20\%$  for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.

**NOTE:** For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD shall be used for sample values greater than ten times

the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.

4. For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be  $\pm 10\%$ . The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.
  5. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.
- E. Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

**TABLE I**

<b>Aqueous Method Detection Limits(MDL), Empirical Laboratories' Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>				
<b>Mercury by EPA 245.1, 7470A, SOW 4.1 &amp; 5.2</b>	<b>AQUEOUS MDL(ug/L)</b>	<b>AQUEOUS ERL(ug/L)</b>	<b>AQUEOUS CRQL ILMO 4.1 (ug/L)</b>	<b>AQUEOUS CRQL ILMO 5.2 (ug/L)</b>
<b>Mercury</b>	0.08	0.20	0.2	0.2

**TABLE 2**

<b>ANALYTE</b>	<b>WAVELENGTH</b>
<b>Mercury</b>	<b>253.7</b>

## XII. CORRECTIVE ACTIONS

### A. INSTRUMENT RELATED

1. ICV(QCS for 245.1)- second source not within  $\pm 10\%$ .
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within  $\pm 20\%$  for SW846 and  $\pm 10\%$  for (245.1,  $\pm 5\%$  for initial IPC and  $+ 10\%$  for subsequent IPCs)
  - a. If the problem is with the solution.
    - i. Reprepare, obtain new stock if necessary.
  - b. If the problem is with the calibration.
    - i. Recalibrate through analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.
      - a. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
      - b. If the CCV was biased low, the previous ten samples must be reanalyzed.

### B. DIGESTION RELATED

1. The preparation blank less than  $<1/2$  RL or  $\pm RL/CRDL$  for common contaminants (DOD) and  $\pm RL/CRDL$  for others and CLP.
  - a. If the problem is with the instrument or stannous chloride.
    - i. Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.
  - b. If the problem is with the digestion.

- i. All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.
2. LCS not within control limits ( or  $\pm 20\%$ ,  $\pm 15\%$  for **245.1** ).
    - a. If the problem is with the instrument.
      - i. Reanalyze when instrument is in control if further sample bottles are available.
    - b. Is the problem is with the digestion.
      - i. If biased low, associated samples must be redigested.
      - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

### C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm 20\%$ 
  - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm 25\%$  ( **$\pm 20\%$  for DOD projects**)
  - i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
  - ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.
3. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

### XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### **XIV. REFERENCES**

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 7470A*
2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B*
3. *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

#### **XV. DEFINITIONS**

1. Refer to SOP-431 for common definitions.

#### **ADDENDUM FOR USEPA SOW ILM05.2**

1. The CCV concentration must be different from the ICV.
2. The same CCV shall be used throughout analysis for an SDG.
3. Calibration standards must be within 5% of the standard concentration.
4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is  $\pm 30\%$ .
5. Spike samples at 1 ug/L for water.

**ANALYST DATA REVIEW CHECKLIST**

<b>Sample Number(s):</b>
<b>Batch Number(s):</b>
<b>Method: 7470A ( 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 LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____

- 11. Analytical results are correct and complete. \_\_\_\_\_
- 12. The appropriate SOP's have been used and followed. \_\_\_\_\_
- 14. "Raw data" including all manual integration's have been correctly interpreted. \_\_\_\_\_
- 15. "Special" sample preparation and analytical requirements have been met. \_\_\_\_\_
- 16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete. \_\_\_\_\_

Comments on any "No" response:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

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**METALS ANALYSIS**

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**BY INDUCTIVELY COUPLED PLASMA-  
ATOMIC EMISSION SPECTROMETRY (ICP-  
AES) TECHNIQUE**

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**METHODS 200.7, ( SW846) 6010B, (SW846)  
6010C, (SM 19<sup>th</sup> Edition 2340B) Hardness  
Calculation, (USEPA CLP) ILMO 4.1 (NJDEP  
does not accept CLPILM 04.1 after June, 2003)  
Addendum for USEPA CLPILM 05.2**

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**SOP NUMBER:**

**SOP-105**

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**REVISION NUMBER:**

**15**

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**APPROVED BY:**

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**SECTION MANAGER**

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**TECHNICAL DIRECTOR**

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**EFFECTIVE DATE:**

**02/22/09**

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**DATE OF LAST REVIEW:**

**05/08/09**

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## ICP METHOD SOP

**References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19<sup>th</sup> Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2**

### I. SCOPE AND APPLICATION

- A. Inductively Coupled Argon Plasma (ICAP) determines trace elements in solution. **We use the ICP to determine the concentration of the following metals: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V and Zn.** All matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.
- B. **Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.** Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

### II. SUMMARY OF METHOD

- A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

- B. This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured.

Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based iTEVA software.

- C. ICP's primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.
- D. It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium internal standard cannot be used.)

### III. SAMPLE HANDLING AND PRESERVATION

- A. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).
- B. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. Since the most concentrated acid matrix allowed for direct disposal down an acid sink is a ratio of 20:1, the samples must be diluted with 1 part water to 2 parts sample prior to pouring down the sink while the tap water is running.
- C. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.
- Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO<sub>3</sub> prior to metal determination.

- Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.
- Standard Methods 19<sup>th</sup> Edition Method 3030C prepares groundwaters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO<sub>3</sub>, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.
- Method 3050B prepares waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

#### IV. INTERFERENCES

- A. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.
3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
4. When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2 from the method, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments

may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

5. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.
7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
8. When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples

analyzed is such they do not contain concentrations of the interfering elements at  $\pm$  one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

- B. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.
- C. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.
- D. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration

accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

## **V. SAFETY**

- A. Normal accepted laboratory safety practices should be followed while performing this analysis.
  - 1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
  - 2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
  - 3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

## **VI. EQUIPMENT/APPARATUS**

- A. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- B. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- C. Radio frequency generator compliant with FCC regulations: Thermo Scientific or equivalent.
- D. Argon gas supply – Liquid Argon
- E. Class A volumetric flasks
- F. Class A volumetric pipettes
- G. Analytical balance - capable of accurate measurement to a minimum of three significant figures (.001gm): Mettler model AE100
- H. Variable Eppendorf Pipettes 1000 $\mu$ L; 5000 $\mu$ L

## **VII. REAGENTS AND STANDARD PREPARATION**

### **A. Notes**

1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

## **B. REAGENTS**

1. Hydrochloric acid (concentrated), HCl.
2. Nitric acid (concentrated), HNO<sub>3</sub>.

## **C. STANDARDS**

### **1. Matrix**

- a. All standards contain 2% HNO<sub>3</sub> and 5% HCl.

### **2. Storage**

- a. The standards are stored at room temperature in 500 mL Teflon bottles.

### **3. Traceability**

- a. All records shall be maintained on all reference materials within Element. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique Element number that is recorded on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date in Element. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations,

volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

#### 4. Calibration standards

- a. All standards have an acid matrix of 2% HNO<sub>3</sub> and 5% HCl and should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs).
- b. STD-1 is the calibration blank: Reagent grade water **matrix matched as in (a) above. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**
- c. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.
- e. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.
- f. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- g. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- h. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- i. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- j. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

- k Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- l Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- m Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- n Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- o Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- p Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- q Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- r Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- s Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- t Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- u Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- v Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

- w. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- x. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- y. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- z. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- aa. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- bb. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- cc. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- dd. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- ee. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- ff. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- gg. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

## 5. Calibration and Calibration Verification standards

- a. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.
- b. The CRI solution is analyzed to check the accuracy of the instrument down near the contract required detection limits (CRDL). It is analyzed in conjunction with the interference check sample. The sample is prepared from a purchased solution which contains 120 µg/mL Sb, 100 µg/mL Co and V, 80 µg/mL Ni, 50 µg/mL Cu, 40 µg/mL Zn, 30 µg/mL Mn, 20 µg/mL As, Cr, Ag and Tl, 10 µg/mL Be, Cd and Se along with 6 µg/mL Pb. 500 µ/L of the solution is diluted to 500 mL. This solution is stable for 6 months.
- hh. The interference check solutions ( ICSA and ICSAB ) are prepared to contain known concentrations of interfering elements that will provide an adequate test of the IECs. A solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the ICSA. The ICSAB is prepared by diluting 100x a solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly.
- d. Reporting Limit Standard- Prepared 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B diluted to 100 ml with 2% HNO<sub>3</sub> and 5% HCL matrix , mix well. Solution stable for 3 months

## 6. Digestion standards

- a. The Laboratory control sample ( LCS ) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3. 25 mL of HCl and 10 mL of HNO<sub>3</sub> are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the LCS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier. The LCS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. Note: The analysis of Molybdenum is not a routine procedure but a project-specific requirement. A customized LCSW mix must be prepared to contain this target analyte.
- b. The solid Laboratory Control Sample (Soil) (LCSS) is prepared by weighing up 1.0 g of teflon chips and spiking using the same spiking

solutions used to spike the sample matrix. This standard is given a unique identifier i.e. LCSS(date prepared)A,B,C etc.

- c. The spiking solutions are prepared as follows:
1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier. Add 0.050 mL (0.20 mL for soil samples) of CLP-CAL-1 solutions A and B, and 0.0125 mL (0.05 mL for soil samples) of CLP-CAL-2 and 3 to 50 mL of sample (1gram of sample for soils) for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.
  2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:  
2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.
  3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:  
0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

## VIII. CALIBRATION AND ASSOCIATED QA/QC

- A. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- B. Operating conditions - **The instrument settings can be found in method file.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
- C. Autopeak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. (**See SOP-106, ICP Instrument Operation**) Flush the system with 2% HNO<sub>3</sub> / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ( $r \geq 0.998$ ). If a three point calibration curve is not required for the client samples being analyzed Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.
- D. Before beginning the sample run, analyze the Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ( $\pm 10\%$ ) for 200.7 ( $\pm 5\%$ ) and ICB ( $< \pm MDL$  or  $\pm RL/CRDL$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $> 2xMDL$** ) first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within  $\pm 20\%$  for DOD projects and  $\pm 30\%$  for samples analyzed for 6010C. Then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition.
- E. For CLP projects, verify the validity of the curve in the region of 2x the contract required detection limit ( CRDL ) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB

criteria:  $< \pm\text{MDL}$  or  $\pm\text{RL/CRDL}$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $>2x\text{MDL}$ , beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Results should be within  $\pm 20\%$ . Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

- F. Verify the interelement and background correction factors at the beginning and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB criteria:  $< \pm\text{MDL}$  or  $\pm\text{RL/CRDL}$  for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected  $>2x\text{MDL}$ , beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check solution A and AB. Results should be within  $\pm 20\%$  of the true value for ICSAB. **For ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes  $< 2x\text{MDL}$ .**(CRI, ICSA and ICSAB required at the end for CLP projects only).
- G. *When analyzing samples associated with North Carolina or with DOD QSM Ver. 3 work, a solution containing analytes at their reporting limit must be analyzed prior to sample analysis. The concentrations must be within 20% DOD( 20 or 30% depending on project) of their true values to be acceptable.*
- Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.
- H. The instrument must be calibrated once every 24 hours if performing straight CLP work.
- I. Instrument Autosampler Report example:

#### **Calibration Rack(used by instrument software to insert QC)**

- 1) STD 1-blank
- 2) Low Cal
- 3) Mid Cal
- 4) Ba @ 5000 ppb
- 5) QC5
- 6) QC 21
- 7) NAK 100
- 8) QC3

#### **Sample Sequence RACK 1**

- 1) Al IEC-(readback)

- 2) Fe IEC-(readback)
- 3) ICV
- 4) ICB-initial
- 5) RL-reporting limit standard
- 6) Ba@ 5000 ppb (readback)
- 7) QC5
- 8) NAK High-(readback)
- 9) QC 21 High-(readback)
- 10) Salt Cal at 500 ppm (readback)
- 11) Rinse
- 12) CRI-0
- 13) ICAS-0
- 14) ICASB-0
- 15) Rinse
- 16) CCV 1A
- 17) CCB 1A
- 18) Preparation Blank (*Batch #* BLK-1)
- 19) Laboratory Control Sample (*Batch #* BS-1)
- 20) Sample 1
- 21) Sample 2
- 22) Sample 3
- 23) Sample 4
- 24) Sample 5
- 25) Sample 6
- 26) Sample 7
- 27) Sample 8
- 28) CCV 1B
- 29) CCB 1B
- 30) Sample 9
- 31) Sample 10
- 32) Sample 11
- 33) Sample 12
- 34) Sample 13
- 35) Sample 14
- 36) Sample 15
- 37) Sample 16
- 38) Sample 17
- 39) Sample 18
- 40) CCV 2A
- 41) CCB 2A
- 42) Sample 19
- 43) Sample 20
- 44) Sample matrix spike (*batch#* MS-1)
- 45) Sample matrix spike duplicate (*batch#* MSD-1)
- 46) Sample post digestion spike (*batch#* PS-1)
- 47) Sample serial dilution (*batch#* SRD-1)
- 48) CRI-1

- 49) ICSA-1
- 50) ICSAB-1
- 51) Rinse
- 52) CCV 2B
- 53) CCB 2B
- 54) Preparation Blank (*batch#* BLK-1)
- 55) Laboratory Control Sample (*batch#* BS-1)
- 56) Sample 1
- 57) Sample 2
- 58) Sample 3
- 59) Sample 4
- 60) Sample 5

## **RACK 2**

- 1) Sample 6
- 2) Sample 7
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

## **IX. PROCEDURE**

- A. Once the instrument has been calibrated, begin the analysis of samples.
- B. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**
- C. Flush the system with 2% HNO<sub>3</sub> / 5% HCl for at least 1 minute before the analysis of each sample.
- D. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7,  $\pm 10\%$  of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and RL are flagged as estimated with a "B" flag.**
- E. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

- The results of the CCV are to agree within 10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from replicate ( minimum of two integrations ). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.
- The results of the calibration blank (this is not the method/preparation blank) are to agree within  $< \pm\text{MDL}$ (SW-846 Method 6010B), and  $3 \times \text{IDL}$  or CRDL for CLP, for **DOD QSM Ver. 3 no analytes detected >2xMDL**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank  $< 1/10$  the concentration of the action level of interest, and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).
- Total hardness is reported from HNO<sub>3</sub> preserved sample. The final concentration is calculated from the calcium and magnesium results as follows:

$$\text{Ca mg/L} \times 2.5 + \text{Mg mg/L} \times 4.1 = \text{total Hardness in mg/L as CaCO}_3$$

- F. Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## X. CALCULATIONS

- A. The instrument will generate data results in mg/L or µg/L ( labeled appropriately). Each result represents an average of three individual readings per metal channel.
- B. For aqueous samples, if a post/predigestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.
- C. For solid samples, if a postdigestion dilution is performed , the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ ( ug/g or mg/kg )} = IR * DF * FED / SM$$

SR	=	Sample result
IR	=	Instrument result ( $\mu\text{g/L}$ )
DF	=	Dilution factor ( post digestion )
FED	=	Final volume of digestate ( L )
SM	=	Sample mass digested( g )

## **XI. QUALITY CONTROL**

### **A. Daily**

1. See sections VIII and IX above.

### **B. Quarterly**

1. Linear range standards must be analyzed at a frequency no less than once every three months. The linear range standard represents the second standard required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-5% of true value.
2. The interelement correction factors ( IEC ) should be verified at the time the linear range standards are analyzed.
3. IDL's if CLP work required.

### **C. Digestion**

1. All quality control data should be maintained and available for easy reference or inspection.
2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank, sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than  $\pm \frac{1}{2}$  RL (USACE) or  $\pm RL/CRDL$  for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified.
3. Employ a minimum of one laboratory control sample ( LCS ) for aqueous samples or one teflon chip spiked sample per sample batch to verify the digestion procedure. These LCSs are taken through the same digestion/preparation steps as the sample being tested. The control limits

are  $\pm 15\%$  method 200.7 - aqueous and soil samples or  $\pm 20\%$  for all other methods aqueous and soil samples. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be redigested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

#### D. Sample

1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are 20% RPD (if both are  $>5x$  CRDL) or  $\pm$  the CRDL (if either are  $<5x$  CRDL).
2. Analyze one spiked sample and spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within  $\pm 25\%$  of the true value ( **$\pm 20\%$  for DOD projects**). If not, a post digestion spike should be analyzed.
3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of  $\pm 20\%$  RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than five times the contract required detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.
4. *The following should be analyzed with each preparation batch containing a matrix spike.*
  - Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within  $\pm 10\%$  of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
  - Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value and is required if the pre-digestion matrix

spike (low-level only for CLH) is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

**E. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:**

**TABLE I**

<b>Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 &amp; OLM05.2 Contract Required Quantitation Limits (CRQL)</b>								
Analytes by EPA 200.7,3005A/30 50A- 6010B SOW 4.1 & 5.2	AQUEOUS MDL (ug/L)	AQUEOUS ERL (ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
<b>Silver</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>	<b>0.20</b>	<b>2.0</b>	<b>2</b>	<b>2</b>
<b>Aluminum</b>	<b>50</b>	<b>200</b>	<b>200</b>	<b>200</b>	<b>10</b>	<b>40</b>	<b>40</b>	<b>40</b>
<b>Arsenic</b>	<b>3.0</b>	<b>10</b>	<b>10</b>	<b>15</b>	<b>0.6</b>	<b>2.0</b>	<b>2</b>	<b>3</b>
<b>Barium</b>	<b>5.0</b>	<b>200</b>	<b>200</b>	<b>200</b>	<b>1.0</b>	<b>40</b>	<b>40</b>	<b>40</b>
<b>Beryllium</b>	<b>1.0</b>	<b>5.0</b>	<b>5</b>	<b>5</b>	<b>0.20</b>	<b>1.0</b>	<b>1</b>	<b>1</b>
<b>Calcium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>20</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Cadmium</b>	<b>1.0</b>	<b>5.0</b>	<b>5</b>	<b>5</b>	<b>0.20</b>	<b>1.0</b>	<b>1</b>	<b>1</b>
<b>Cobalt</b>	<b>5.0</b>	<b>50</b>	<b>50</b>	<b>50</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>
<b>Chromium</b>	<b>2.0</b>	<b>10</b>	<b>10</b>	<b>10</b>	<b>0.40</b>	<b>2.0</b>	<b>2</b>	<b>2</b>
<b>Copper</b>	<b>4.0</b>	<b>25</b>	<b>25</b>	<b>25</b>	<b>0.40</b>	<b>5.0</b>	<b>5</b>	<b>5</b>
<b>Iron</b>	<b>30</b>	<b>100</b>	<b>100</b>	<b>100</b>	<b>1.0</b>	<b>20</b>	<b>20</b>	<b>20</b>
<b>Potassium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Magnesium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Manganese</b>	<b>1.0</b>	<b>15</b>	<b>15</b>	<b>15</b>	<b>0.20</b>	<b>3.0</b>	<b>3</b>	<b>3</b>
<b>Sodium</b>	<b>1000</b>	<b>5000</b>	<b>5000</b>	<b>5000</b>	<b>40</b>	<b>1000</b>	<b>1000</b>	<b>1000</b>
<b>Nickel</b>	<b>3.0</b>	<b>40</b>	<b>40</b>	<b>40</b>	<b>1.0</b>	<b>8.0</b>	<b>8</b>	<b>8</b>
<b>Lead</b>	<b>1.5</b>	<b>5.0</b>	<b>3</b>	<b>10</b>	<b>0.60</b>	<b>2.0</b>	<b>0.6</b>	<b>2</b>
<b>Selenium</b>	<b>3.0</b>	<b>10</b>	<b>5</b>	<b>35</b>	<b>0.60</b>	<b>2.0</b>	<b>1</b>	<b>7</b>
<b>Antimony</b>	<b>5.0</b>	<b>60</b>	<b>60</b>	<b>60</b>	<b>1.0</b>	<b>12</b>	<b>12</b>	<b>12</b>
<b>Thallium</b>	<b>3.0</b>	<b>10</b>	<b>10</b>	<b>25</b>	<b>0.60</b>	<b>2.0</b>	<b>2</b>	<b>5</b>
<b>Vanadium</b>	<b>5.0</b>	<b>50</b>	<b>50</b>	<b>50</b>	<b>1.0</b>	<b>10</b>	<b>10</b>	<b>10</b>
<b>Zinc</b>	<b>5.0</b>	<b>20</b>	<b>20</b>	<b>60</b>	<b>1.0</b>	<b>4.0</b>	<b>4</b>	<b>12</b>

**TABLE 2**

<b>METAL</b>	<b>WAVELENGTH</b>
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

**XII. CORRECTIVE ACTIONS****A. INSTRUMENT RELATED**

1. ICV not within  $\pm 10\%$  or  $\pm 5\%$  for 200.7
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate through analysis of appropriate standards and recheck ICV.

2. ICB not  $\pm$ MDL or within  $\pm$  3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**
  - a. Is the problem with the solution?
    - i. Reprepare
  - b. Is the problem with the calibration?
    - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.
3. Check standards not within  $\pm$  5%
  - a. Is the problem with the solution?
    - i. Repour, reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
4. CRI not within  $\pm$  20% (Internal QC, only required for CLP work).
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
5. ICSA metals not present are not less than the CRDL for that metal, **for ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL.**
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
6. ICSAB not within  $\pm$  20%
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
7. CCV not within  $\pm$  10%
  - a. Is the problem with the solution?
    - i. Reprepare or obtain new stock.
  - b. Is the problem with the calibration?
    - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified. Note: CLH data must

always be reanalyzed back to the last compliant CCV and not qualified.

8. CCB not  $\pm$ MDL or within  $\pm$  3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**
  - a. Is the problem with the solution?
    - i. Reprepare
  - b. Is the problem with the calibration?
    - i. Apply SW846 guidance. (See Section IX-E for additional guidance). Note: CLH data must always be reanalyzed back to the last compliant CCB and not qualified.

## B. DIGESTION RELATED

1. Preparation blank not within  $\pm$  1/2 RL and  $\pm$  RL for common contaminants USACE or RL/CRDL for other or CLP
  - a. Is the problem with the instrument?
    - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  - b. Is the problem with the digestion?
    - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be redigested or the data must be qualified on the final report.
2. LCS not within control limits
  - a. Is the problem with the instrument?
    - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  - b. Is the problem with the digestion?
    - i. If biased low, associated samples must be redigested.
    - ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

## C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within  $\pm$ 20% (if both are >5X CRDL) or  $\pm$  the CRDL ( if either are <5X CRDL).
  - a. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within  $\pm$ 25% ( **$\pm$  20% for DOD projects**)
  - a. Is the analyte level in the sample greater than 4X the spiking level?
    - i. If yes, the spike recovery is not evaluated.
    - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.
3. When required, post digestion spike analysis recovery not within  $\pm$ 15%.

- a. The associated sample data must be qualified on the final report.
  - b. For USACE analysis by MSA is required.
4. Serial dilution analysis percent difference not within  $\pm 10\%$ 
    - a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
      - i. If no, the serial dilution data can not be evaluated.
      - iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

### **XIII. WASTE DISPOSAL and POLLUTION PREVENTION**

Please see Waste Disposal SOP-405 for instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

### **XIV. REFERENCES**

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B and Method 6010C*
2. *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 200.7; APX-B*
3. *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

Refer to SOP-431 for common environmental laboratory definitions.

### **Addendum for USEPA CLPILM 05.2**

1. The control limit for the ICSA is at 20% or  $\pm$ CRQL whichever is greater.
2. Preparation codes are required in the digestion log See SOW Exhibit B for a listing of these codes with definitions.
3. The CRQL check standard is run at the concentration of the respective CRQLs. For a listing of CRQL for this SOW see Exhibit C. Several of the metals concentration levels have changed.
4. The spiking level for CLP ILM 05.2 is at 50 ug/L for selenium. All other spike levels remain the same as in SOW ILM 04.1.
5. The CCV shall be analyzed at a different concentration then the ICV (at or near one-half of the calibration standard concentration).
6. The post digestion spike must be analyzed at 2x the indigenous level of the sample or two times the CRQL whichever is greater.
7. A Non-prepared MDL study must be analyzed and the results of this study used for MDL reporting when sample volumes are not digested.

### **CHANGES TO FORMS for SOWCLPILM 05.2**

1. Forms must be double-sided
2. A photocopy of the instrument's direct sequential readout shall be included.
3. Undiluted samples must be reported as well as diluted samples.
4. J flags are used in place of B flags when a sample has a concentration less the CRQL but greater then or equal to the MDL.
5. A D flag is used for samples reported from a dilution.
6. All results are reported down to the MDL not the IDL.
7. Preparation codes are used on form 13.

The form for method of standard additions (MSA) has been removed and all subsequent QC has move up one form number in other words form 8 is now serial dilution when it used to be the MSA form,etcetera.

<b>ANALYST DATA REVIEW CHECKLIST Sample Number(s):</b>				
<b>Batch Number(s):</b>				
<b>Method: 6010B ( ICP )</b>				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did LCS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

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Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Second-Level Review: \_\_\_\_\_ Date: \_\_\_\_\_

## **APPENDIX I**

### **Preparation Method for Calibration Standards**

**Analytical Standard Record**  
**Empirical Laboratories, LLC****09E0096**

Description:	LOW CAL	Expires:	07/28/200
Standard Type:	Calibration Stan	Prepared:	04/28/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	05/07/2009 10:00 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	5	mg/L
Antimony	7440-36-0	0.1	mg/L
Arsenic	7440-38-2	0.1	mg/L
Barium	7440-39-3	0.05	mg/L
Beryllium	7440-41-7	0.1	mg/L
Boron	7440-42-8	0.05	mg/L
Cadmium	7440-43-9	0.1	mg/L
Calcium	7440-70-2	1	mg/L
Chromium	7440-47-3	0.1	mg/L
Cobalt	7440-48-4	0.1	mg/L
Copper	7440-50-8	0.1	mg/L
Iron	7439-89-6	5	mg/L
Aluminum	7429-90-5	5	mg/L
Lithium	7439-93-2	0.1	mg/L
Zinc	7440-66-6	0.1	mg/L
Manganese	7439-96-5	0.1	mg/L
Molybdenum	7439-98-7	0.1	mg/L
Nickel	7440-02-0	0.1	mg/L
Potassium	7440-09-7	1	mg/L
Selenium	7782-49-2	0.1	mg/L
Silver	7440-22-4	0.02	mg/L
Sodium	7440-23-5	1	mg/L
Strontium	7440-24-6	0.1	mg/L
Thallium	7440-28-0	0.1	mg/L
Tin	7440-31-5	0.05	mg/L
Titanium	7440-32-6	0.1	mg/L
Vanadium	7440-62-2	0.1	mg/L
Lead	7439-92-1	0.1	mg/L

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC**  
**09E0096**

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09D0300	NAK100ppm	04/16/200	Roger Burr	06/24/200	04/16/2009 13:32 by RBU	1
09D0306	QC21 HIGH	04/16/200	Roger Burr	07/03/200	04/16/2009 14:53 by RBU	1
09E0062	QC5	04/29/200	Roger Burr	07/29/200	05/06/2009 08:43 by RGB	1

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Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC**  
**09E0097**

Description:	MID CAL.	Expires:	08/07/200
Standard Type:	Calibration Stan	Prepared:	05/07/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	05/07/2009 10:22 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC**  
**09E0097****Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0025	SINGLE ELEMENT CALCIUM	02/03/200	Roger Burr	05/13/201	02/03/2009 11:43 by RBU	0.49
09D0297	MAGNESIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	0.49
09D0305	QCS7M	04/16/200	Roger Burr	02/10/201	04/16/2009 14:28 by RBU	1
09D0310	QC21	04/16/200	Roger Burr	05/30/200	04/16/2009 14:57 by RBU	1
09E0058	Aluminum	05/06/200	Roger Burr	04/30/201	05/06/2009 08:30 by RGB	0.09
09E0059	Iron	05/06/200	Roger Burr	04/30/201	05/06/2009 08:32 by RGB	0.09
09E0060	Sodium	05/06/200	Roger Burr	04/30/201	05/06/2009 08:33 by RGB	0.49
09E0061	SN100	05/06/200	Roger Burr	10/29/200	05/06/2009 08:37 by RGB	1

Reviewed By

Date

## Analytical Standard Record

Empirical Laboratories, LLC

09D0302

Description:	QC3	Expires:	07/03/200
Standard Type:	Calibration Stan	Prepared:	04/03/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/07/2009 10:05 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	500	ppm
Magnesium	7439-95-4	100	ppm
Calcium	7440-70-2	500	ppm

## Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0025	SINGLE ELEMENT CALCIUM	02/03/200	Roger Burr	05/13/201	02/03/2009 11:43 by RBU	25
09D0295	SODIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:31 by RBU	25
09D0297	MAGNESIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	5

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC**  
**09E0068**

Description:	NAK100ppm	Expires:	08/06/200
Standard Type:	Calibration Stan	Prepared:	05/06/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/06/2009 10:14 by RGB

Analyte	CAS Number	Concentration	Units
Sodium	7440-23-5	100	ppm
Potassium	7440-09-7	100	ppm
Calcium	7440-70-2	100	ppm

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09D0293	CALCIUM	04/16/200	Roger Burr	01/23/201	04/16/2009 13:24 by RBU	5
09D0295	SODIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:31 by RBU	5
09D0298	POTASSIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:32 by RBU	5

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC****09E0062**

Description:	QC5	Expires:	07/29/200
Standard Type:	Calibration Stan	Prepared:	04/29/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/06/2009 08:43 by RGB

Analyte	CAS Number	Concentration	Units
Silver	7440-22-4	2	ppm
Magnesium	7439-95-4	500	ppm
Iron	7439-89-6	500	ppm
Barium	7440-39-3	5	ppm
Aluminum	7429-90-5	500	ppm

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0019	SINGLE ELEMENT ALUMINUM	02/03/200	Roger Burr	05/13/201	02/03/2009 11:21 by RBU	25
09D0281	IRON	04/16/200	Roger Burr	10/03/201	04/16/2009 13:25 by RBU	25
09D0294	BARIUM	04/16/200	Roger Burr	01/23/201	04/16/2009 13:24 by RBU	0.25
09D0297	MAGNESIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	25
09D0299	SILVER	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	1

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC**  
**09E0099**

Description:	QC21HIGH	Expires:	07/03/200
Standard Type:	Calibration Stan	Prepared:	04/03/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	500	Department:	METALS
Vials:	1	Last Edit:	05/07/2009 11:07 by RGB

Analyte	CAS Number	Concentration	Units
Magnesium	7439-95-4	10	mg/L
Arsenic	7440-38-2	10	mg/L
Beryllium	7440-41-7	10	mg/L
Boron	7440-42-8	5	mg/L
Cadmium	7440-43-9	10	mg/L
Calcium	7440-70-2	10	mg/L
Chromium	7440-47-3	10	mg/L
Cobalt	7440-48-4	10	mg/L
Copper	7440-50-8	10	mg/L
Iron	7439-89-6	10	mg/L
Antimony	7440-36-0	10	mg/L
Lithium	7439-93-2	10	mg/L
Zinc	7440-66-6	10	mg/L
Manganese	7439-96-5	10	mg/L
Molybdenum	7439-98-7	10	mg/L
Nickel	7440-02-0	10	mg/L
Selenium	7782-49-2	10	mg/L
Strontium	7440-24-6	10	mg/L
Thallium	7440-28-0	10	mg/L
Tin	7440-31-5	5	mg/L
Titanium	7440-32-6	10	mg/L
Vanadium	7440-62-2	10	mg/L
Lead	7439-92-1	10	mg/L

Reviewed By

Date

**Analytical Standard Record****Empirical Laboratories, LLC****09E0099****Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0021	SINGLE ELEMENT BORON	02/03/200	Roger Burr	03/04/201	02/03/2009 11:28 by RBU	2.5
09D0288	TIN	04/16/200	Roger Burr	01/31/201	04/16/2009 13:24 by RBU	0.25
09D0307	QC21	04/16/200	Roger Burr	02/15/201	04/16/2009 14:23 by RBU	50

Reviewed By

Date

**Analytical Standard Record****Empirical Laboratories, LLC****09D0516**

Description:	Ba 5000	Expires:	07/14/200
Standard Type:	Calibration Stan	Prepared:	04/14/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	04/22/2009 16:35 by RGB

Analyte	CAS Number	Concentration	Units
Barium	7440-39-3	5	ppm

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09D0294	BARIUM	04/16/200	Roger Burr	01/23/201	04/16/2009 13:24 by RBU	0.05

Reviewed By \_\_\_\_\_

Date \_\_\_\_\_

**Analytical Standard Record**  
**Empirical Laboratories, LLC**

ICV

09E0065

**Parent Standards used in this standard:**

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0025	SINGLE ELEMENT CALCIUM	02/03/200	Roger Burr	05/13/201	02/03/2009 11:43 by RBU	0.49
09D0297	MAGNESIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	0.49
09D0308	QCS7A	04/16/200	Roger Burr	03/30/201	04/16/2009 14:29 by RBU	1
09D0309	QC23	04/16/200	Roger Burr	07/31/201	04/16/2009 14:36 by RBU	1
09E0058	Aluminum	05/06/200	Roger Burr	04/30/201	05/06/2009 08:30 by RGB	0.09
09E0059	Iron	05/06/200	Roger Burr	04/30/201	05/06/2009 08:32 by RGB	0.09
09E0060	Sodium	05/06/200	Roger Burr	04/30/201	05/06/2009 08:33 by RGB	0.49

Reviewed By

Date

**Analytical Standard Record****Empirical Laboratories, LLC****09E0065**

Description:	ICV METALS	Expires:	05/07/200
Standard Type:	Calibration Stan	Prepared:	05/06/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	05/06/2009 09:06 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Phosphate, Total as P	NA	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	5	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Reviewed By

Date

## Analytical Standard Record

Empirical Laboratories, LLC

09E0064

ccv

Parent Standards used in this standard:						
Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
09B0025	SINGLE ELEMENT CALCIUM	02/03/200	Roger Burr	05/13/201	02/03/2009 11:43 by RBU	0.49
09D0297	MAGNESIUM	04/16/200	Roger Burr	09/09/201	04/16/2009 13:23 by RBU	0.49
09D0305	QCS7M	04/16/200	Roger Burr	02/10/201	04/16/2009 14:28 by RBU	1
09D0310	QC21	04/16/200	Roger Burr	05/30/200	04/16/2009 14:57 by RBU	1
09E0058	Aluminum	05/06/200	Roger Burr	04/30/201	05/06/2009 08:30 by RGB	0.09
09E0059	Iron	05/06/200	Roger Burr	04/30/201	05/06/2009 08:32 by RGB	0.09
09E0060	Sodium	05/06/200	Roger Burr	04/30/201	05/06/2009 08:33 by RGB	0.49
09E0061	SN100	05/06/200	Roger Burr	10/29/200	05/06/2009 08:37 by RGB	1

Reviewed By

Date

**Analytical Standard Record**  
**Empirical Laboratories, LLC****09E0064**

Description:	CCV METALS	Expires:	05/07/200
Standard Type:	Calibration Stan	Prepared:	05/06/200
Solvent:	HNO3	Prepared By:	Roger Burr
Final Volume (mls):	100	Department:	METALS
Vials:	1	Last Edit:	05/06/2009 08:52 by RGB

Analyte	CAS Number	Concentration	Units
Manganese	7439-96-5	1	ppm
Antimony	7440-36-0	1	ppm
Arsenic	7440-38-2	1	ppm
Barium	7440-39-3	1	ppm
Beryllium	7440-41-7	1	ppm
Boron	7440-42-8	1	ppm
Cadmium	7440-43-9	1	ppm
Calcium	7440-70-2	50	ppm
Chromium	7440-47-3	1	ppm
Cobalt	7440-48-4	1	ppm
Copper	7440-50-8	1	ppm
Iron	7439-89-6	10	ppm
Lead	7439-92-1	1	ppm
Aluminum	7429-90-5	10	ppm
Magnesium	7439-95-4	50	ppm
Zinc	7440-66-6	1	ppm
Molybdenum	7439-98-7	1	ppm
Nickel	7440-02-0	1	ppm
Potassium	7440-09-7	10	ppm
Selenium	7782-49-2	1	ppm
Silicon	7440-21-3	1	ppm
Silver	7440-22-4	0.5	ppm
Sodium	7440-23-5	50	ppm
Strontium	7440-24-6	1	ppm
Thallium	7440-28-0	1	ppm
Tin	7440-31-5	1	ppm
Titanium	7440-32-6	1	ppm
Vanadium	7440-62-2	1	ppm
Lithium	7439-93-2	1	ppm

Reviewed By

Date

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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.

## 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
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15. Data Analysis and Calculations
16. Method Performance
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## 1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

## 2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. **Detection Limit:** See **Table 1** of this SOP.

## 4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. 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, NaHSO<sub>4</sub> – 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 ( <b>Cat#30265</b> )	20000	25	20	200
Vinyl Acetate ( <b>#3766</b> )	5000	100	80	200
Ketones ( <b>cat#30006</b> )	5000	100	80	200
Liquid mix ( <b>C-349H-07</b> )	2000	100	100	100
Custom mix ( <b>CCS-1037</b> )	5000	50	40	100
Gases ( <b>cat#30042</b> )	2000	100	100	100
Acrolein/Acrylonitrile ( <b>CC2098.10</b> )	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**

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
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

**LABORATORY SAMPLE RECEIVING,**  

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**LOG IN AND STORAGE**  

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**STANDARD OPERATING PROCEDURES**  

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**SOP NUMBER:** **SOP-404**

**REVISION NUMBER:** **12**

**APPROVED BY:**  

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**SECTION MANAGER**

---

**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** **01/05/09**

**DATE OF LAST REVIEW :** **01/05/09**

## 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 French Landing Drive in Nashville, TN.
  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 on the fifth floor. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we often 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. 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, and 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. 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. In addition to notifying the analyst of samples with short holding times, log-in personnel must log this information into a separate record book daily. It is the analysts' responsibility to review this information and initial each page at the end of each day.

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 as soon as possible 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 entered into the Unpreserved Metals Log and a 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 VII]** 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 Blue Air refrigerator in Sample Storage Room: 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 in Sample Storage Room: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator in back BC Laboratory: 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, 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. 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.

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. **Fraction:**

When more than one container is sent with the same preservation/analysis (i.e. Volatiles & Extractables), the sample containers can be grouped together using a fraction code. This is simply an alphabetical notation added to each container to allow analysts to verify the preservative and proper sample volume to use when performing analysis. **This should be in no way used to record the sample volume used during analysis or reporting**, as these codes are entered by log-in personnel on an as needed basis, and do not provide an individual container designation by which to track any given container.

17. **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.

18. **Comments:**

Enter any information that is applicable at the sample level.

19. **Field Analysis:**

Click on field analysis tab and enter field information when provided.

20. **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.

21. *Analyses Comments:*

These comments should be used for any notes that only apply to that particular test code.

22. *RTAT:*

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

23. *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 (0811248-06)
- 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.

**24. 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. Complete the short holding times log book as required. This must be done as early in the day as possible.
- C. 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).
- D. Complete any required CARs.
- E. 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.
- G. 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.
- H. 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.

I. 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. Yellow copy originals or photo copies will be filed by client and kept in the Sample Receiving Room.
- c. Photo copies will be kept in a notebook in numerical order in the Sample Receiving Room.
- d. 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 test's 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>
SRLB #3	pH Paper Calibration
SRLB #4	Tracking of VOC Trip Blanks, Organic Free Water and Chemical Preservation

**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.]*



**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 acid 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 under the table 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.**

**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**

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**TECHNICAL DIRECTOR**

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**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.